

Fig 1

A

Step : 0.050° Cnt Time: 1.000 Sec.
Range: 2.00 - 40.00 (Deg) Cont. Scan Rate : 3.00 Deg/min.

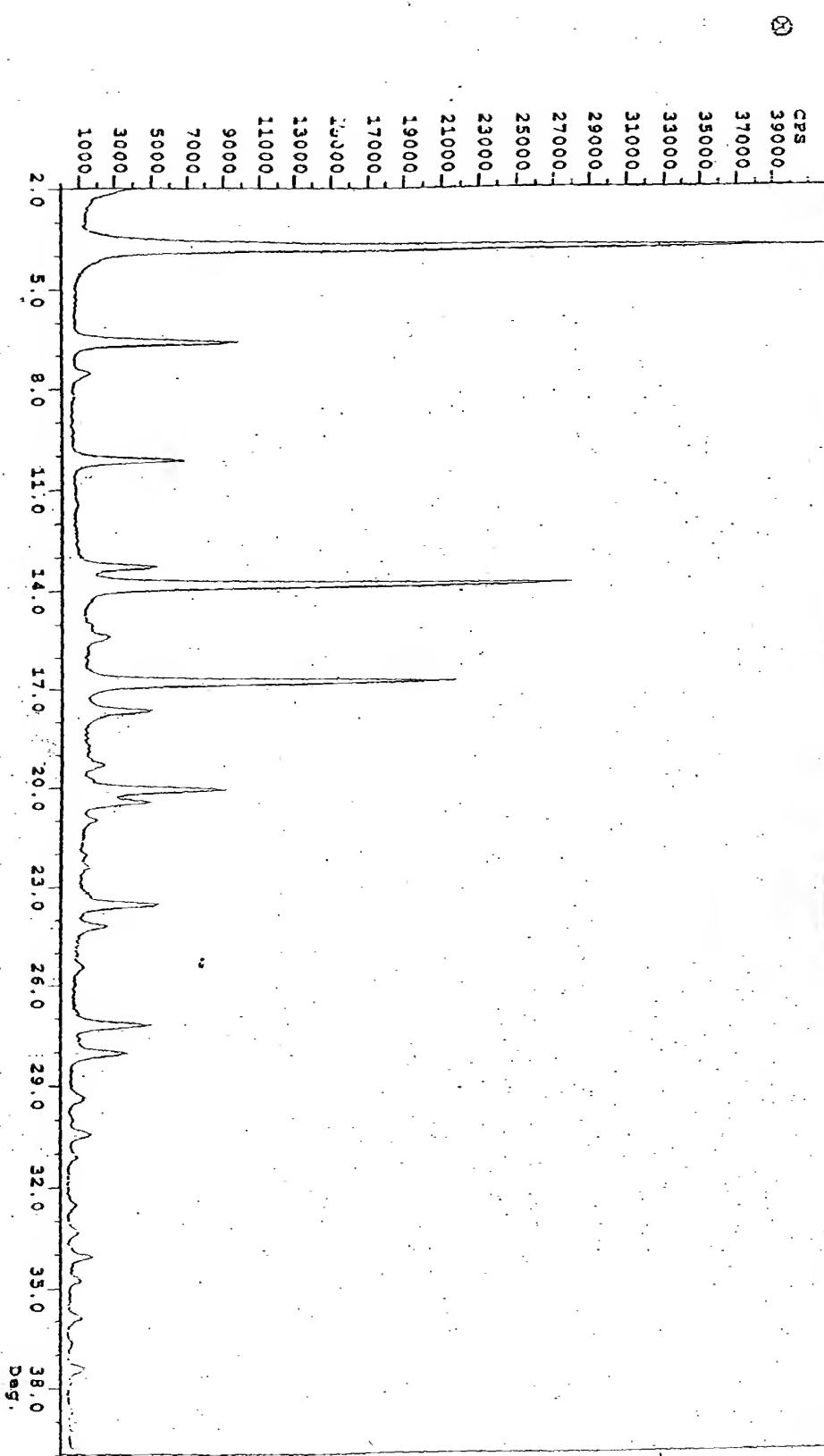


Fig. 2 C

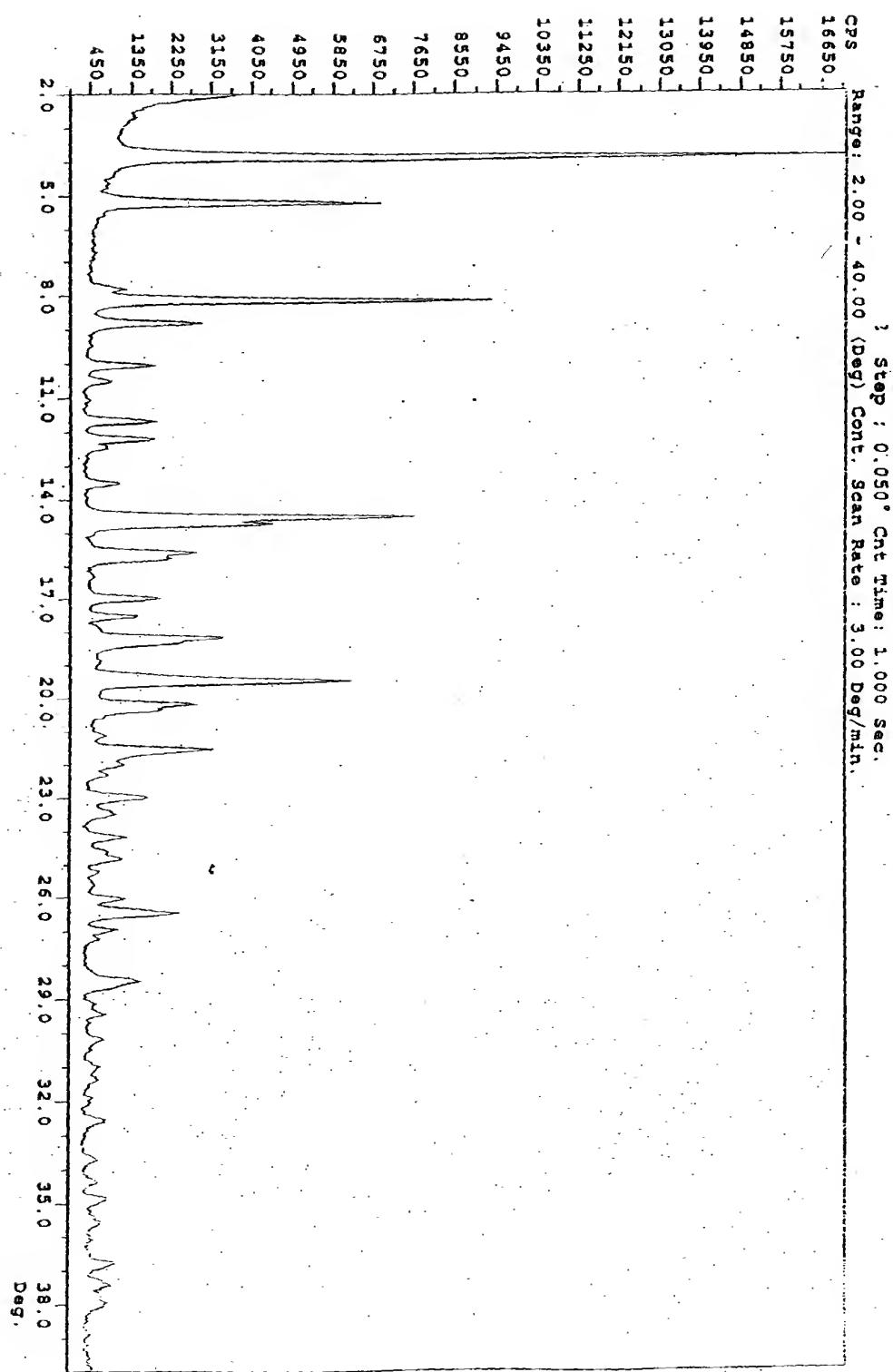


Fig 3 - D

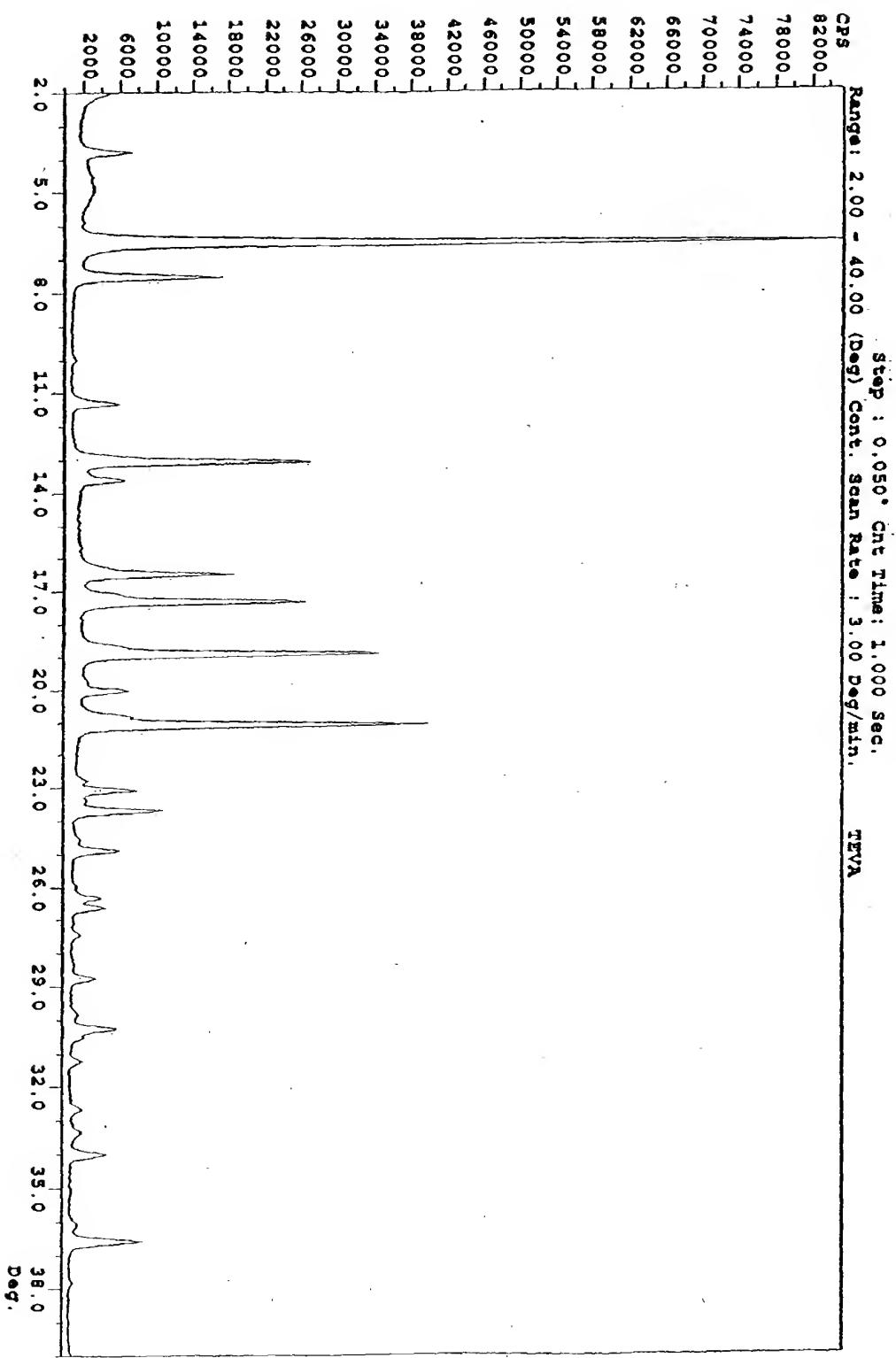


Fig 4 E

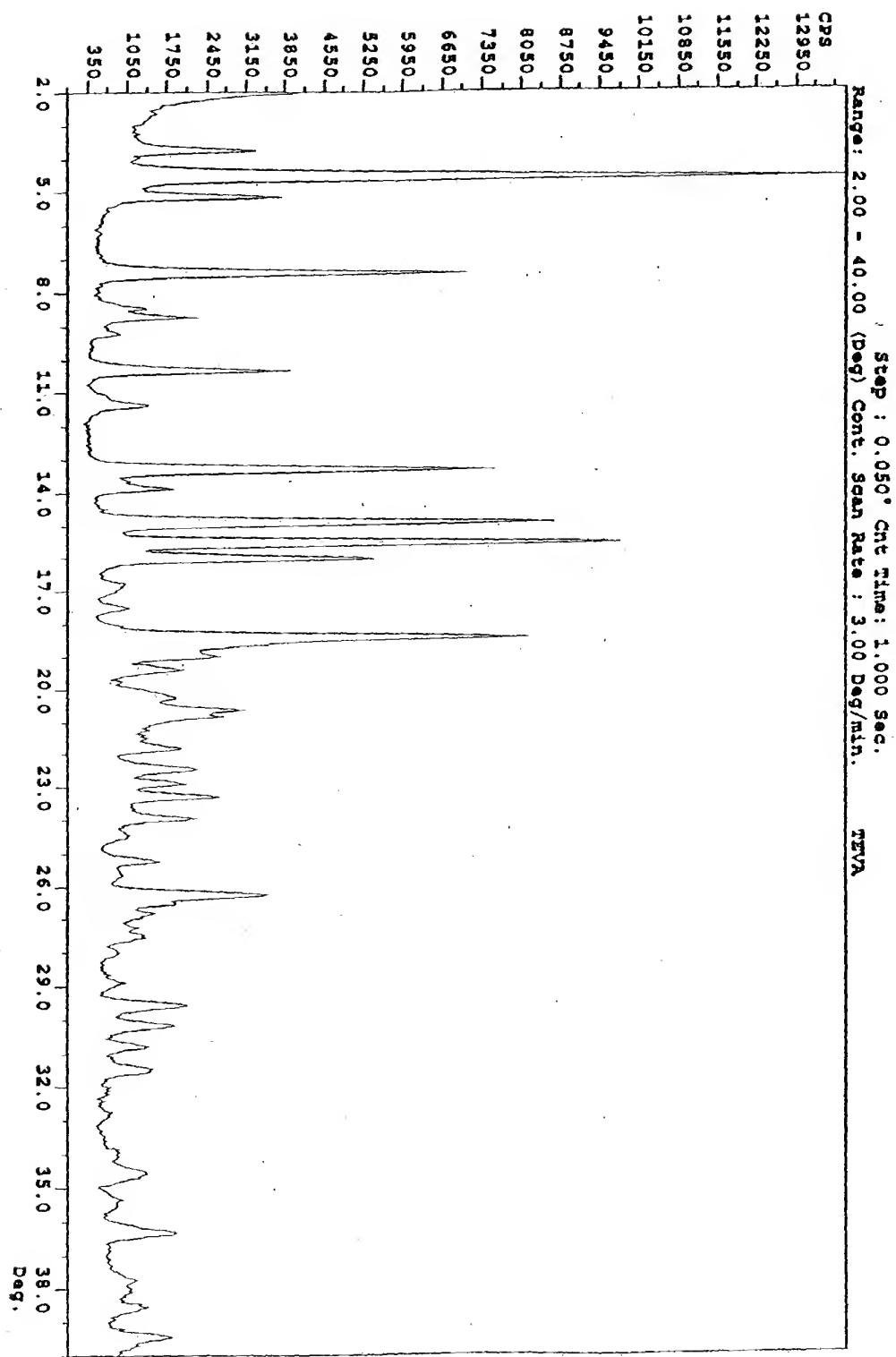


Fig. 5

F

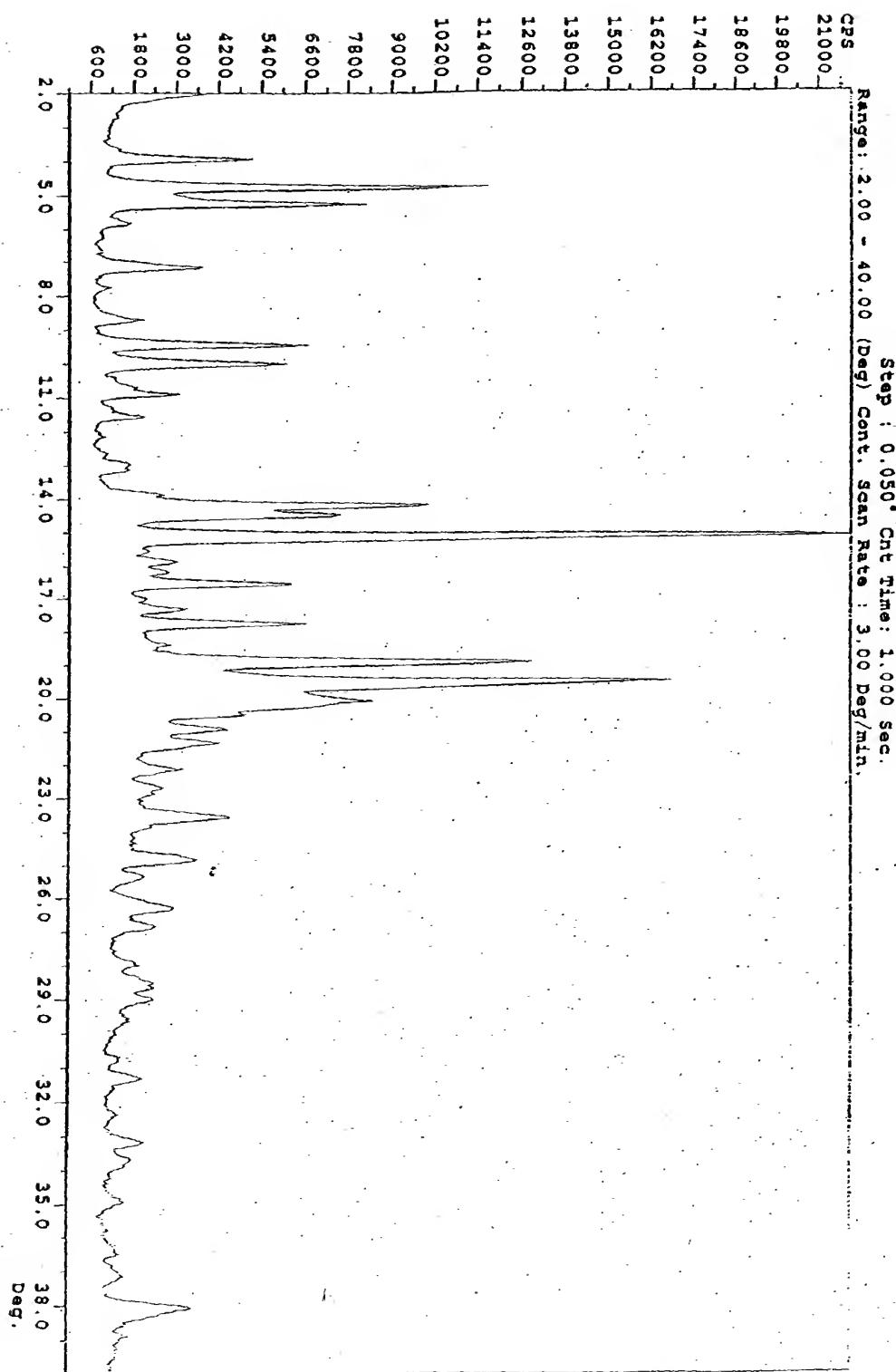


Fig. 6
G

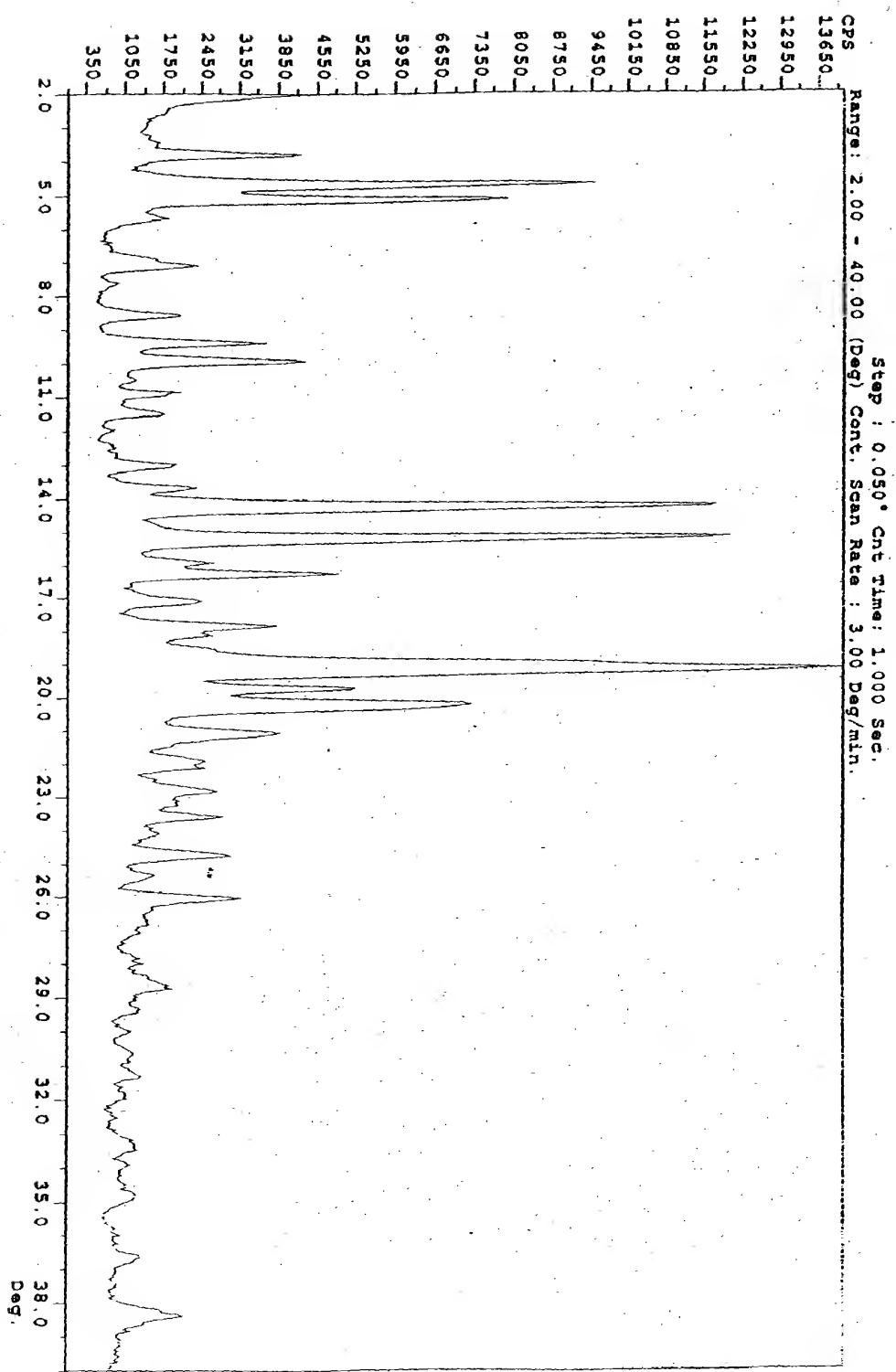


Fig 7 T

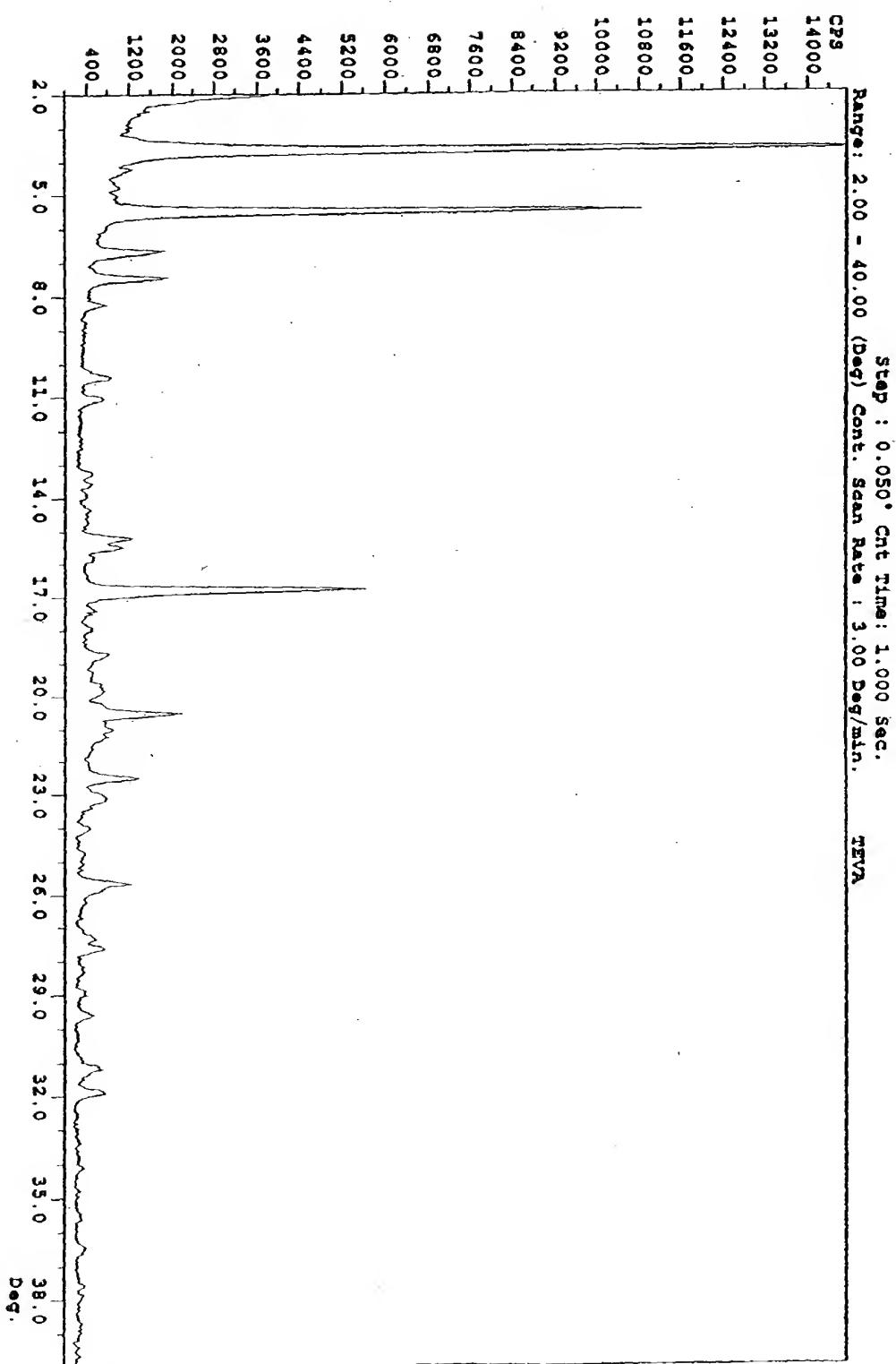


Fig. 8
5

Range: 2.00 - 40.00 (Deg) Step: 0.050° Cnt Time: 1.000 Sec.

Cont. Scan Rate: 3.00 Deg/min.

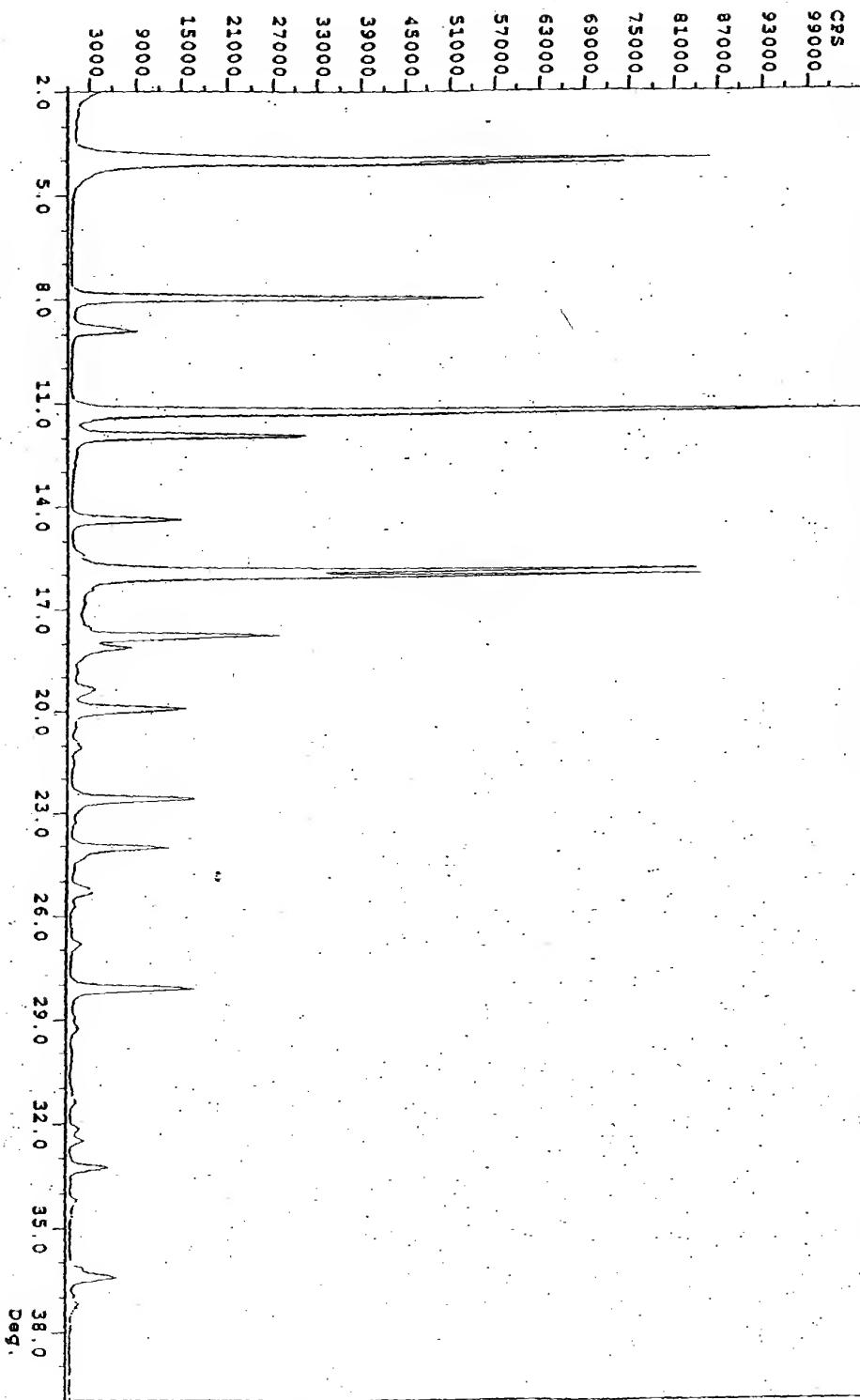


Fig. 9

K

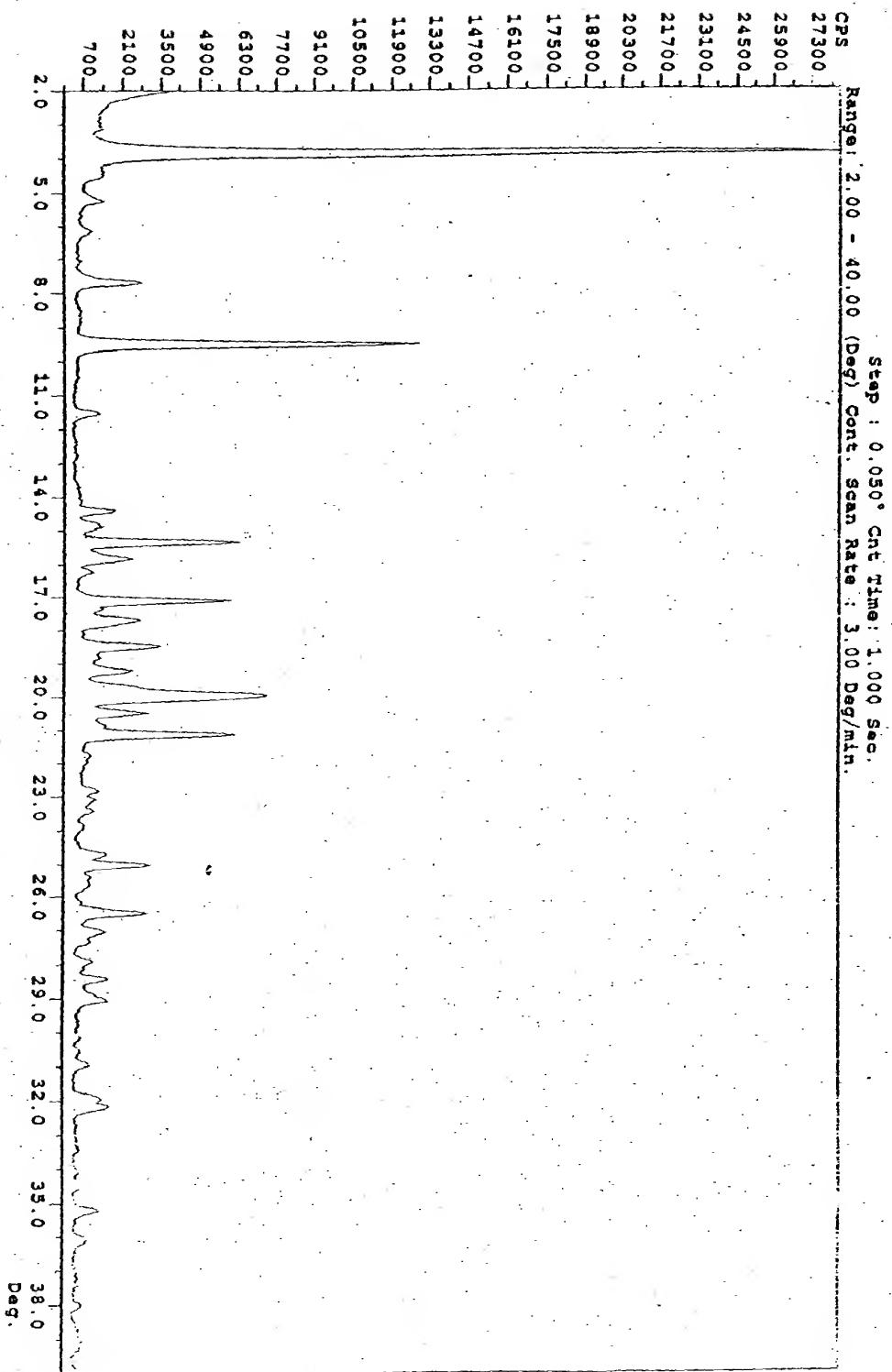


Fig. 10
L

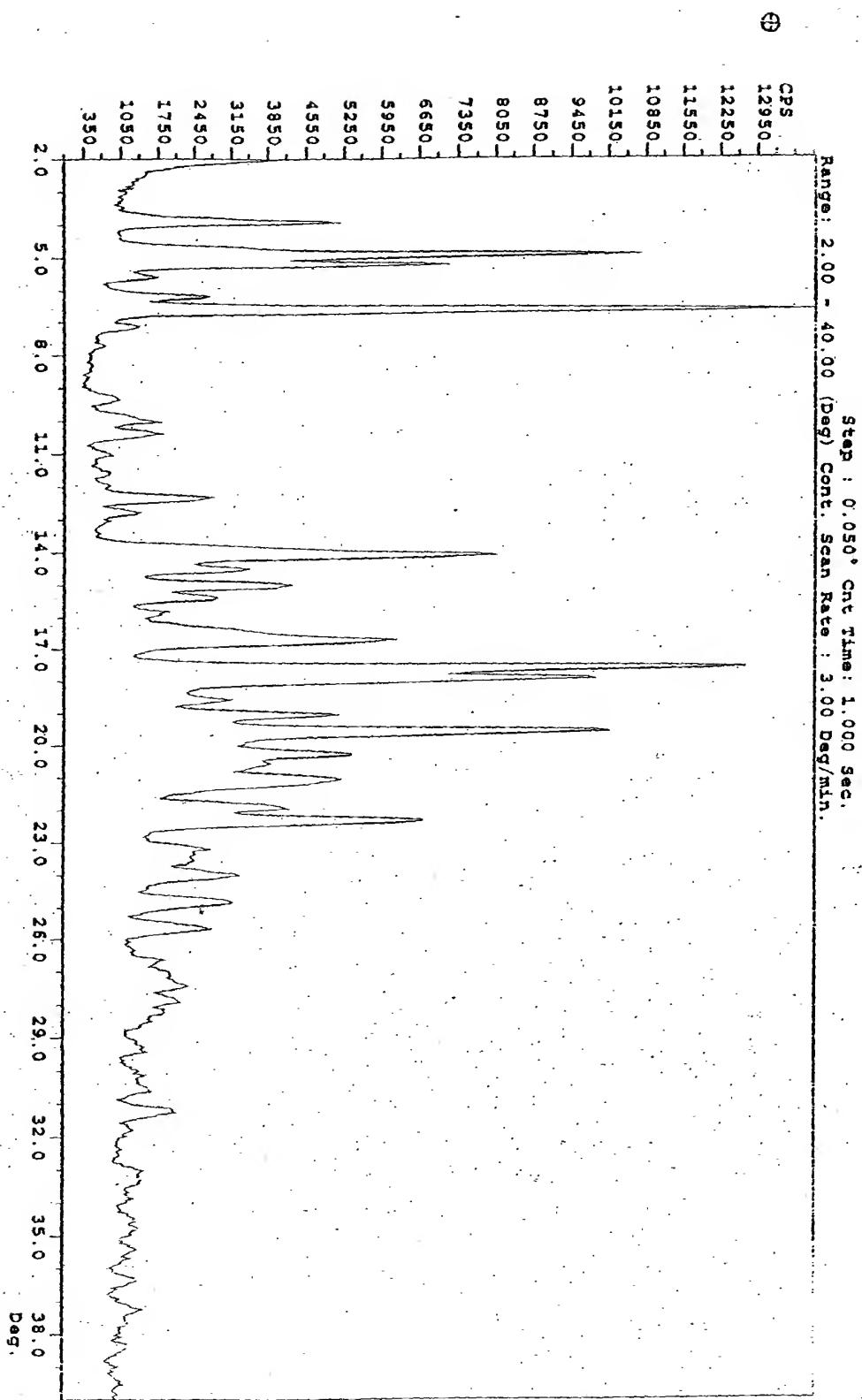


Fig. 11

M

Range: 2.00 - 40.00. Step: 0.050°. Cnt Time: 1.000 Sec.

CPS

7000

6600

6200

5800

5400

5000

4600

4200

3800

3400

3000

2600

2200

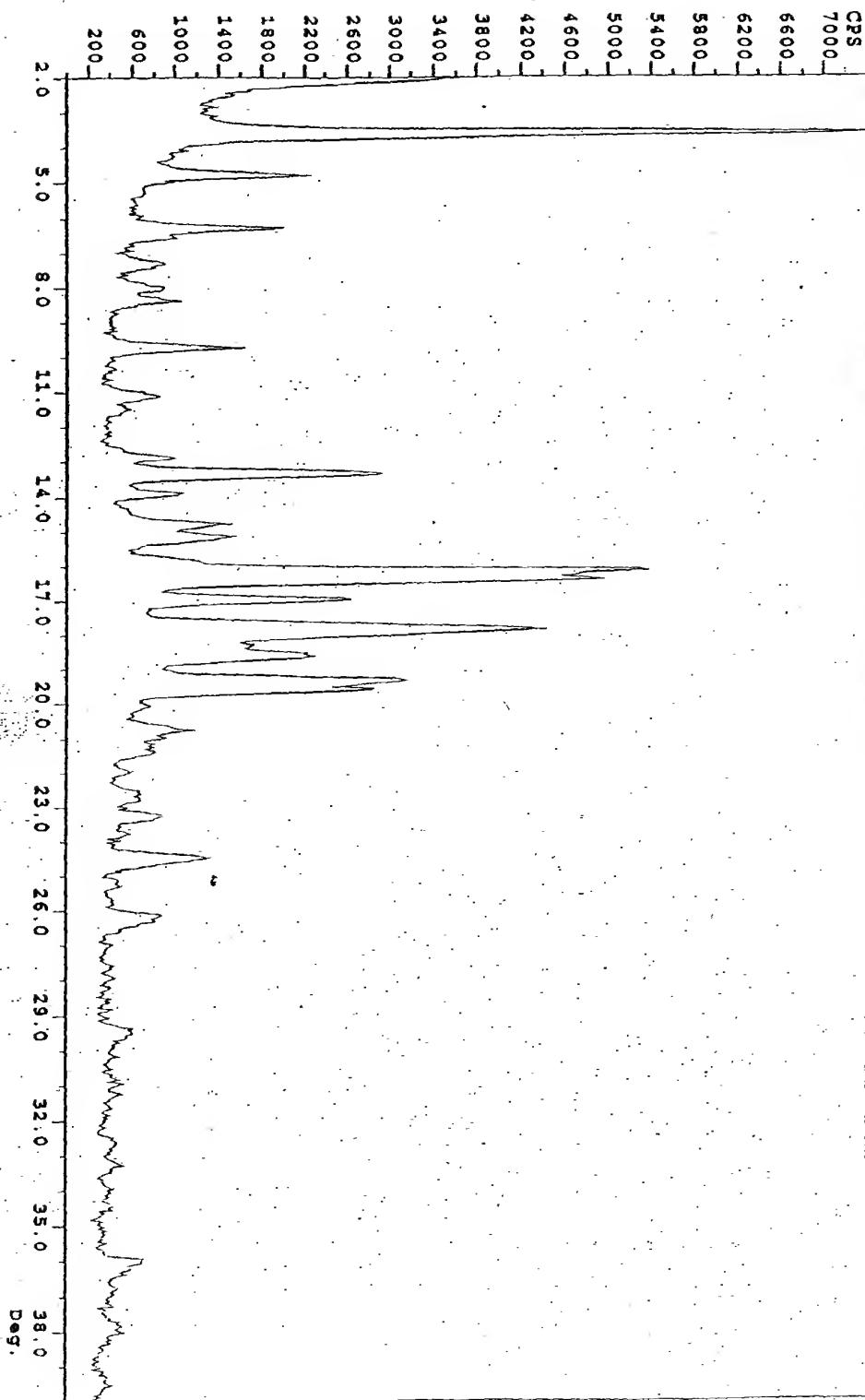
1800

1400

1000

600

200



⊕

Fig. 12

N

Step : 0.050° Cnt Time: 1.000 sec.
Range: 2.00 - 40.00 (Deg) Cont. Scan Rate : 3.00 Deg/min.

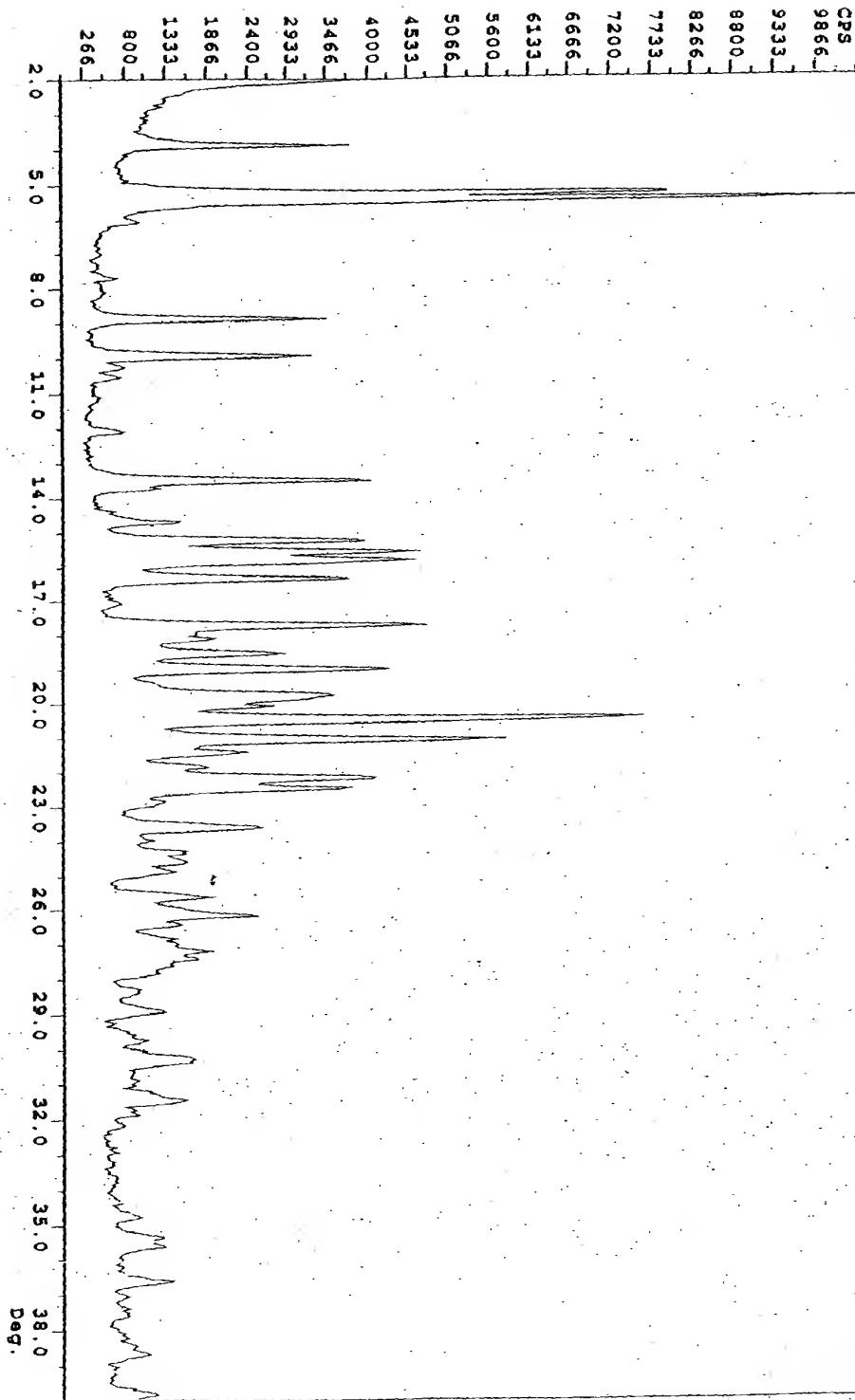
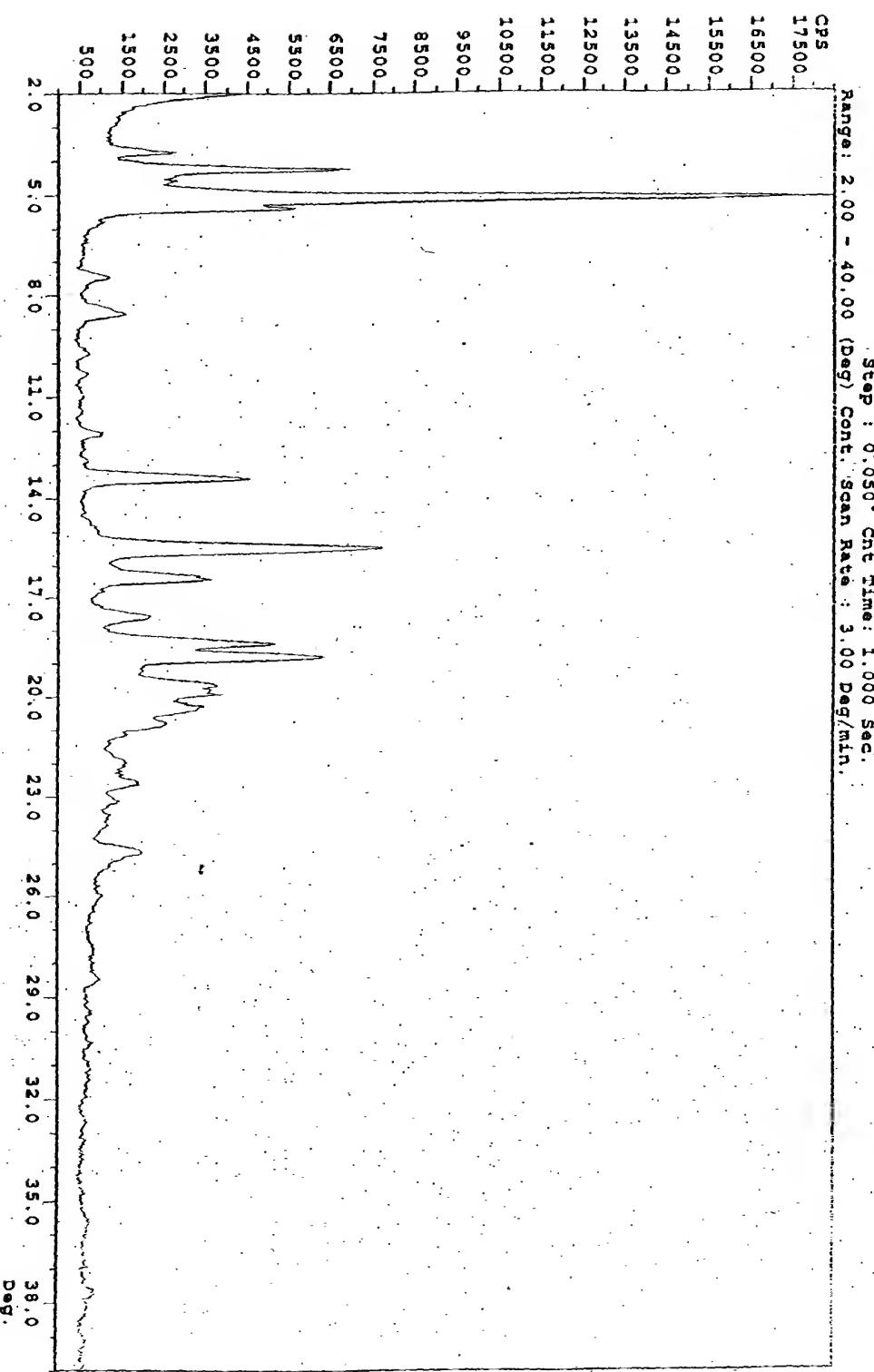


Fig. 13 O



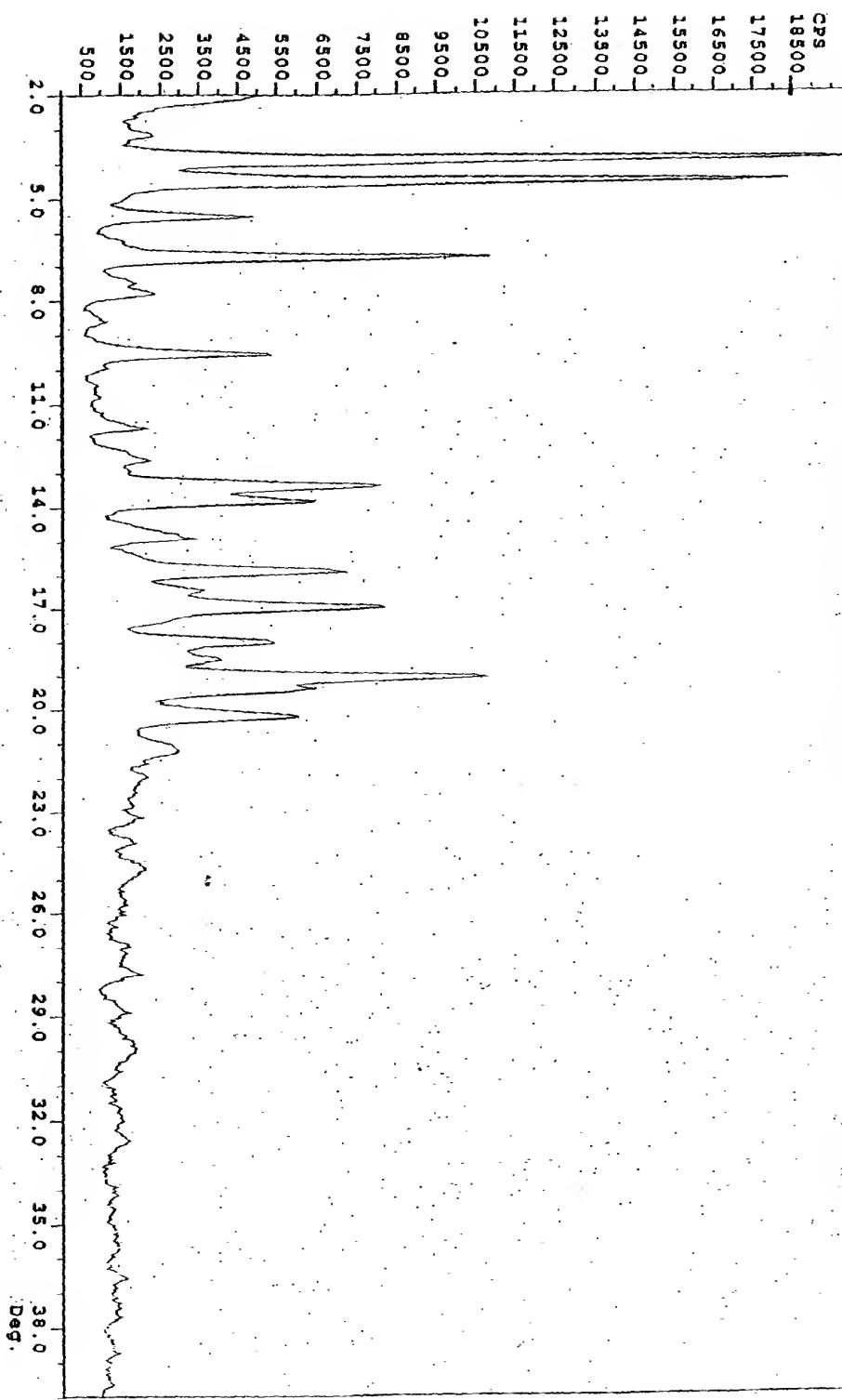
⊗

fig. 14

P

Range: 2.00 - 40.00 (Deg) Step: 0.050° Cnt Time: 1.000 Sec.

Cont. Scan Rate: 3.00 Deg/min.



④

Fig. 15

Q

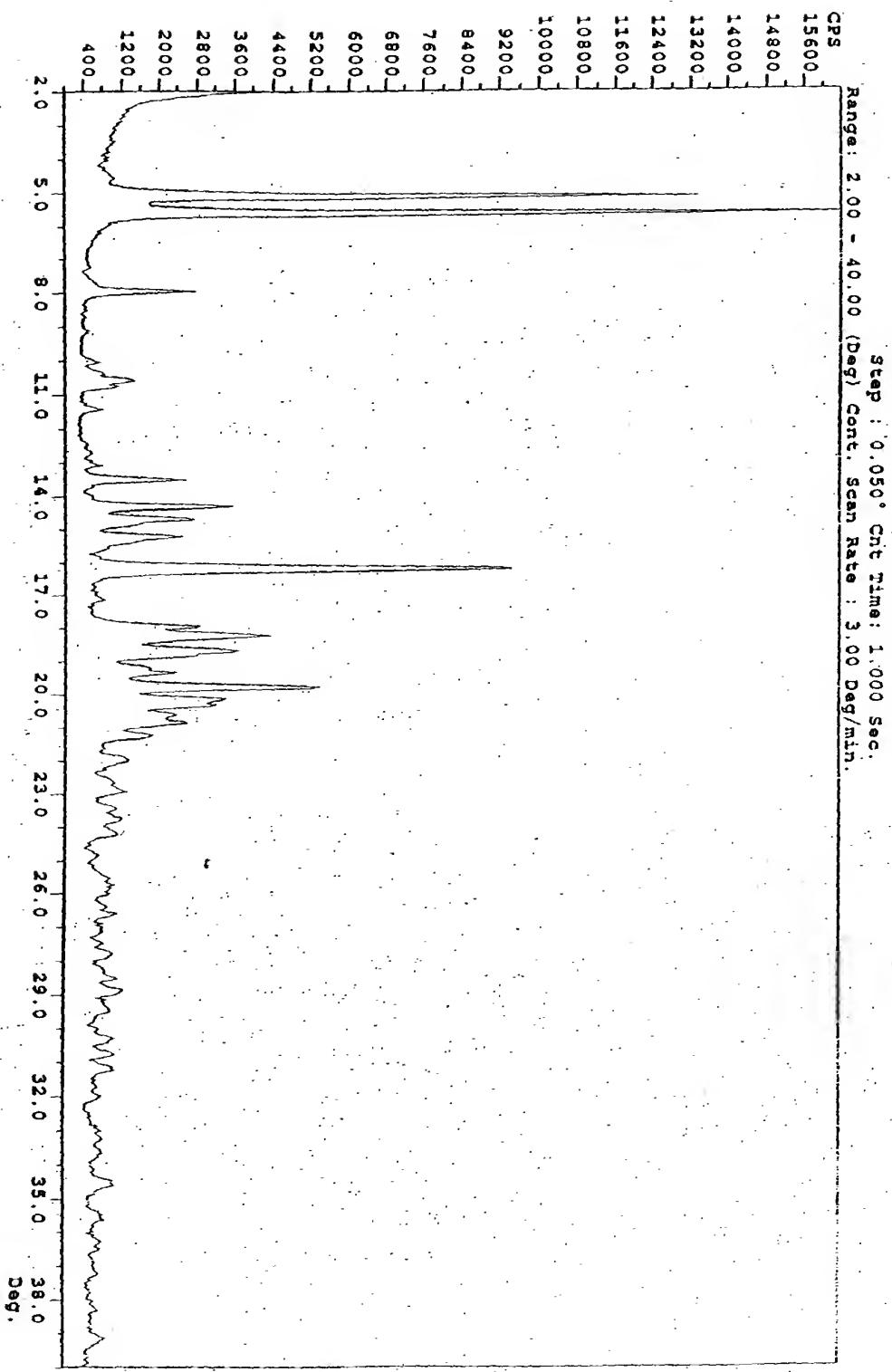
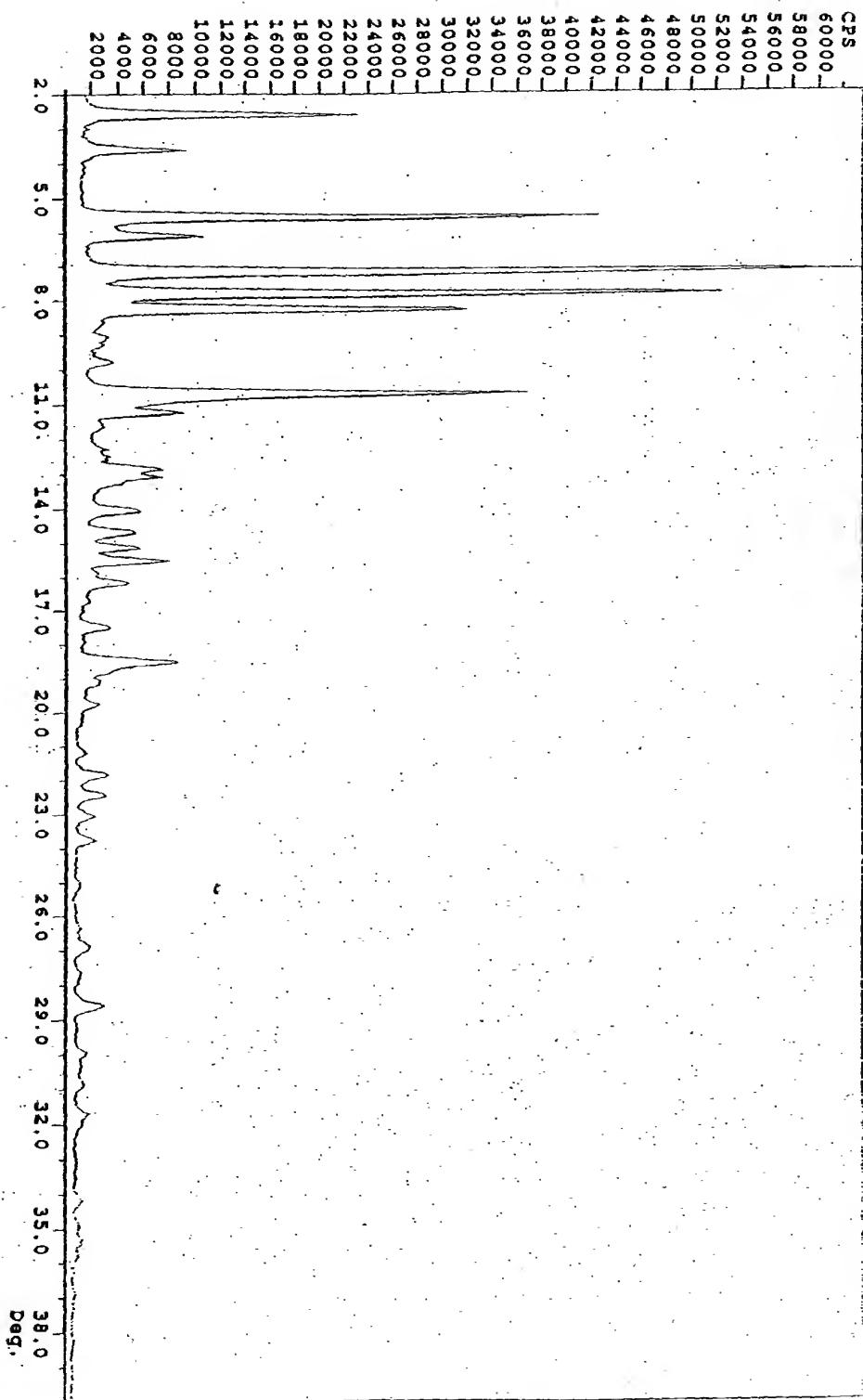


Fig. 16 - T

Range: 2.00 - 40.00 (Deg) Cnt Time: 1.000 Sec.
Step : 0.050° Cont. Scan Rate : 3.00 Deg/min.



④

Fig. 17

U

Range: 2.00 - 40.00 (Deg) Step: 0.050° Cnt Time: 1.000 sec.

CPS

29600

28000

26400

24800

23200

21600

20000

18400

16800

15200

13600

12000

10400

8800

7200

5600

4000

2400

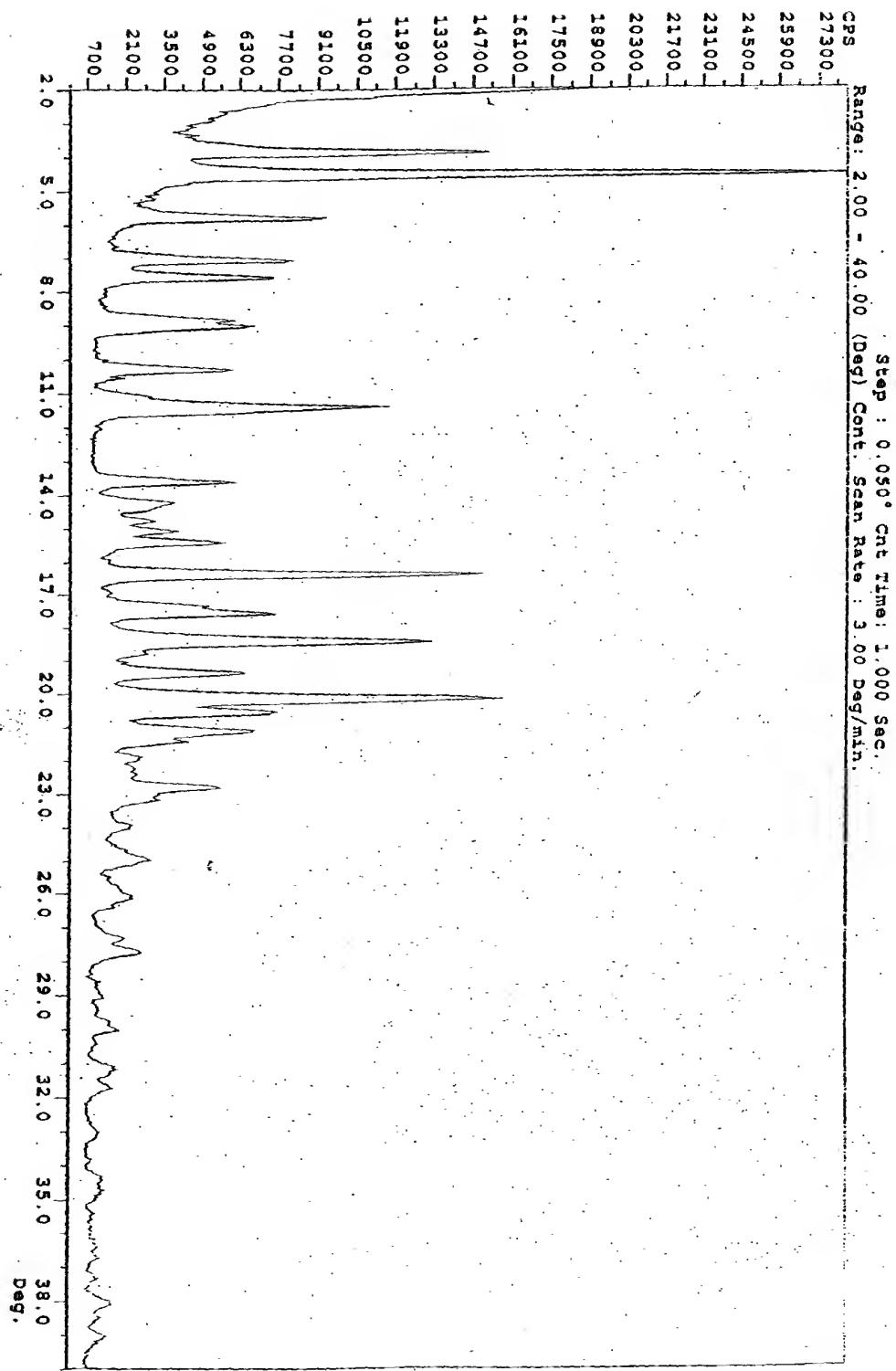
800

2.0 5.0 8.0 11.0 14.0 17.0 20.0 23.0 26.0 29.0 32.0 35.0 38.0

⊕

Fig. 18

✓



⊕

fig 19 r

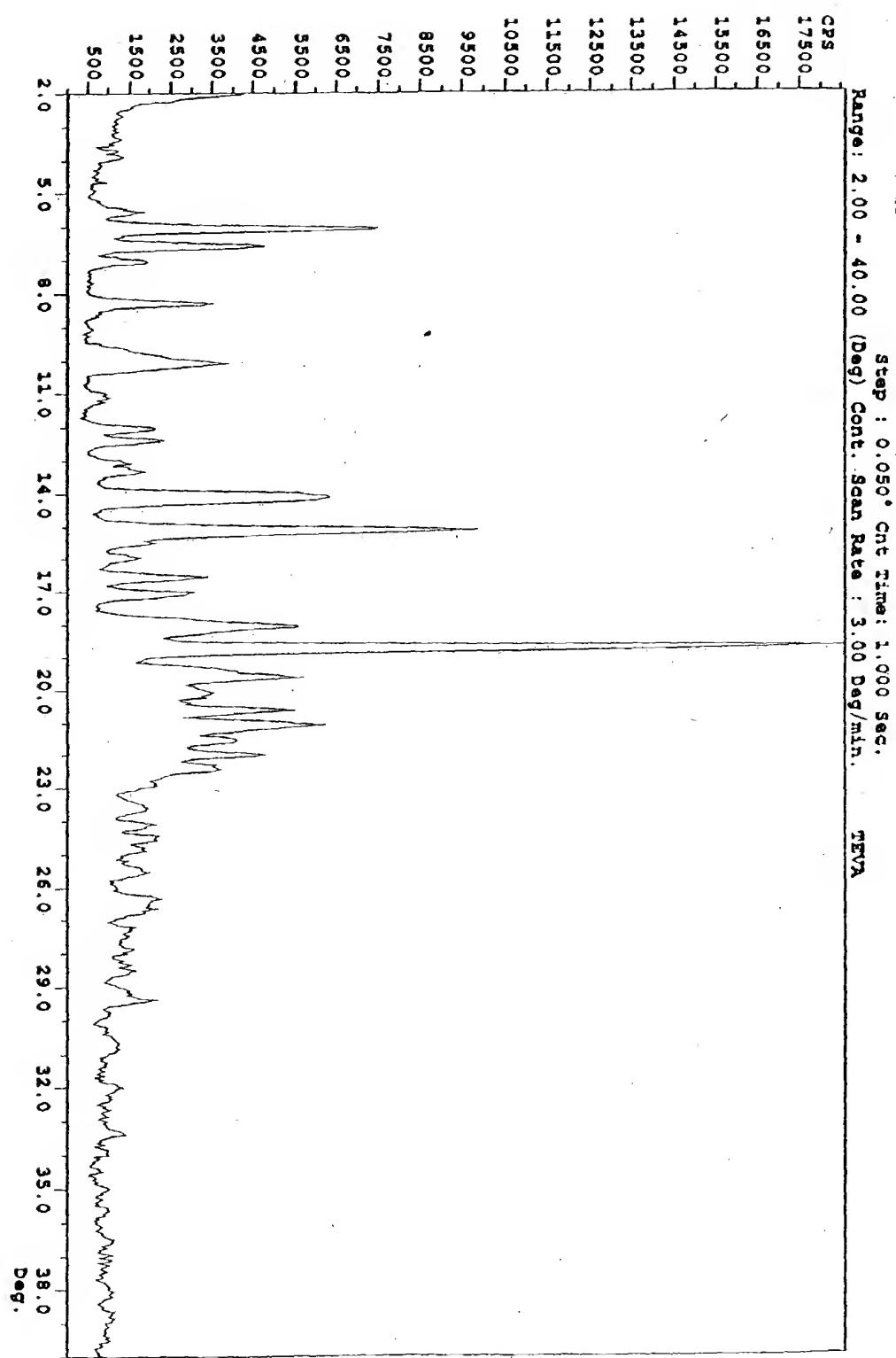


Figure 20 - Natta Linde Form Z

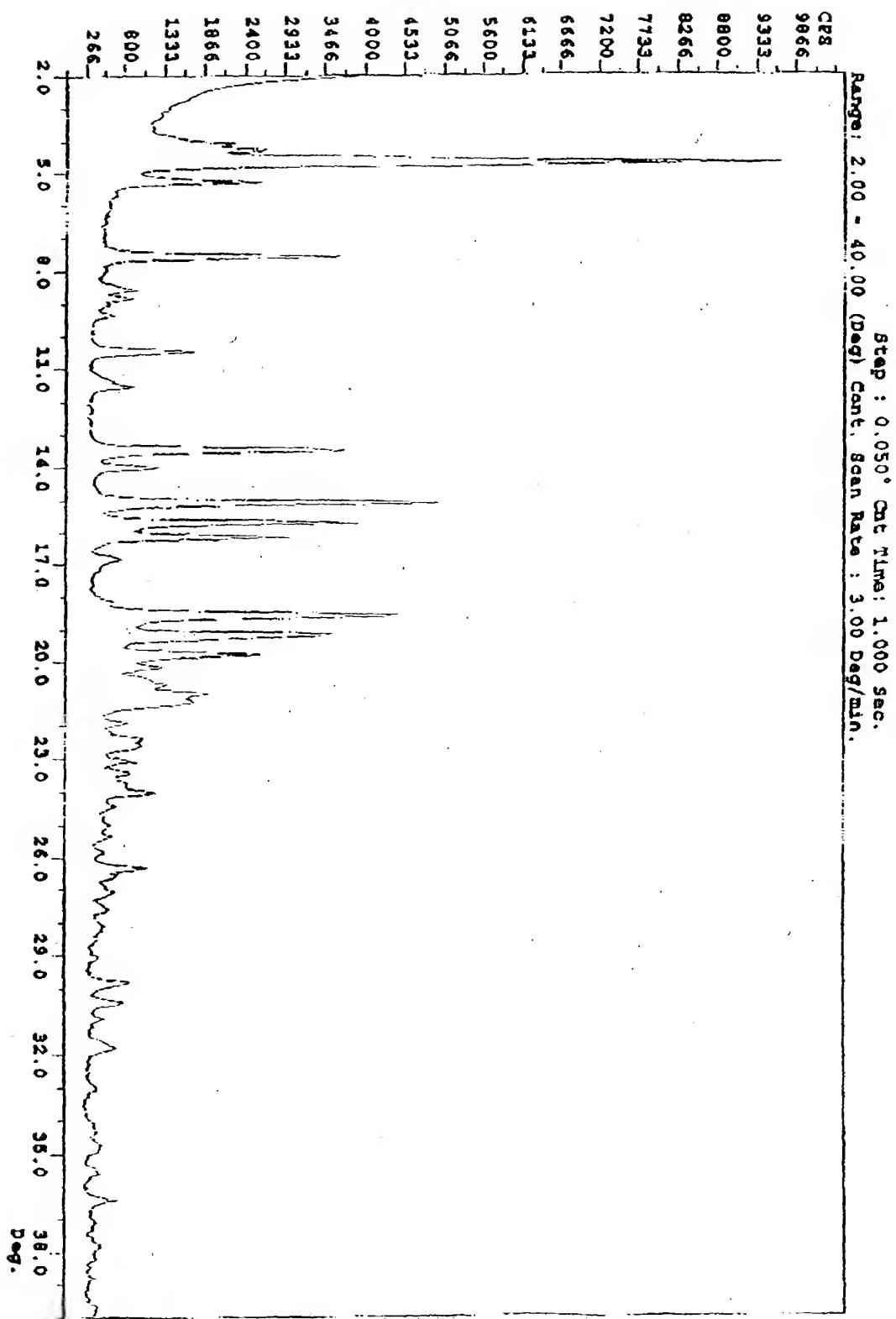


fig 2b x

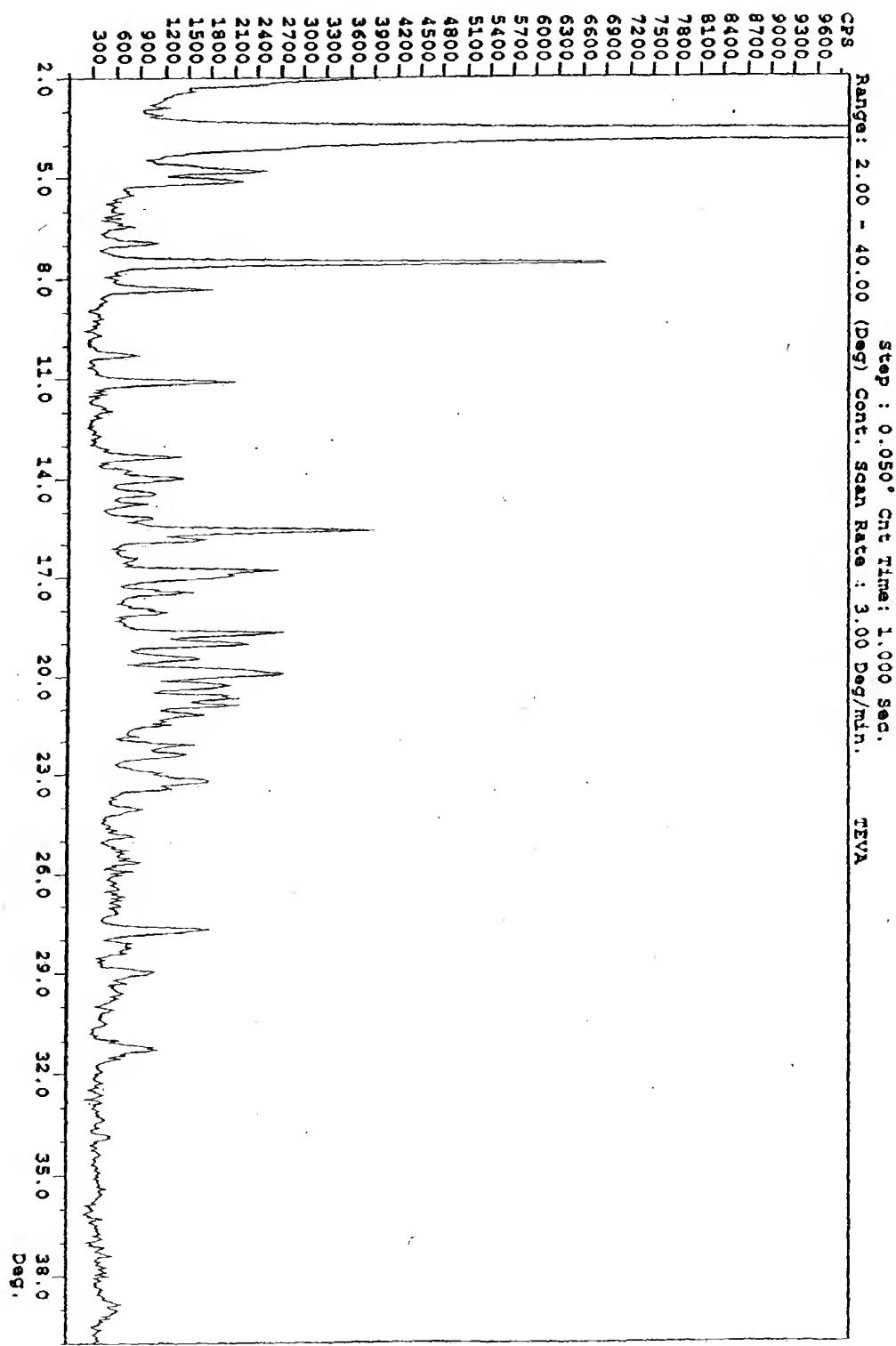


Fig. 2a β

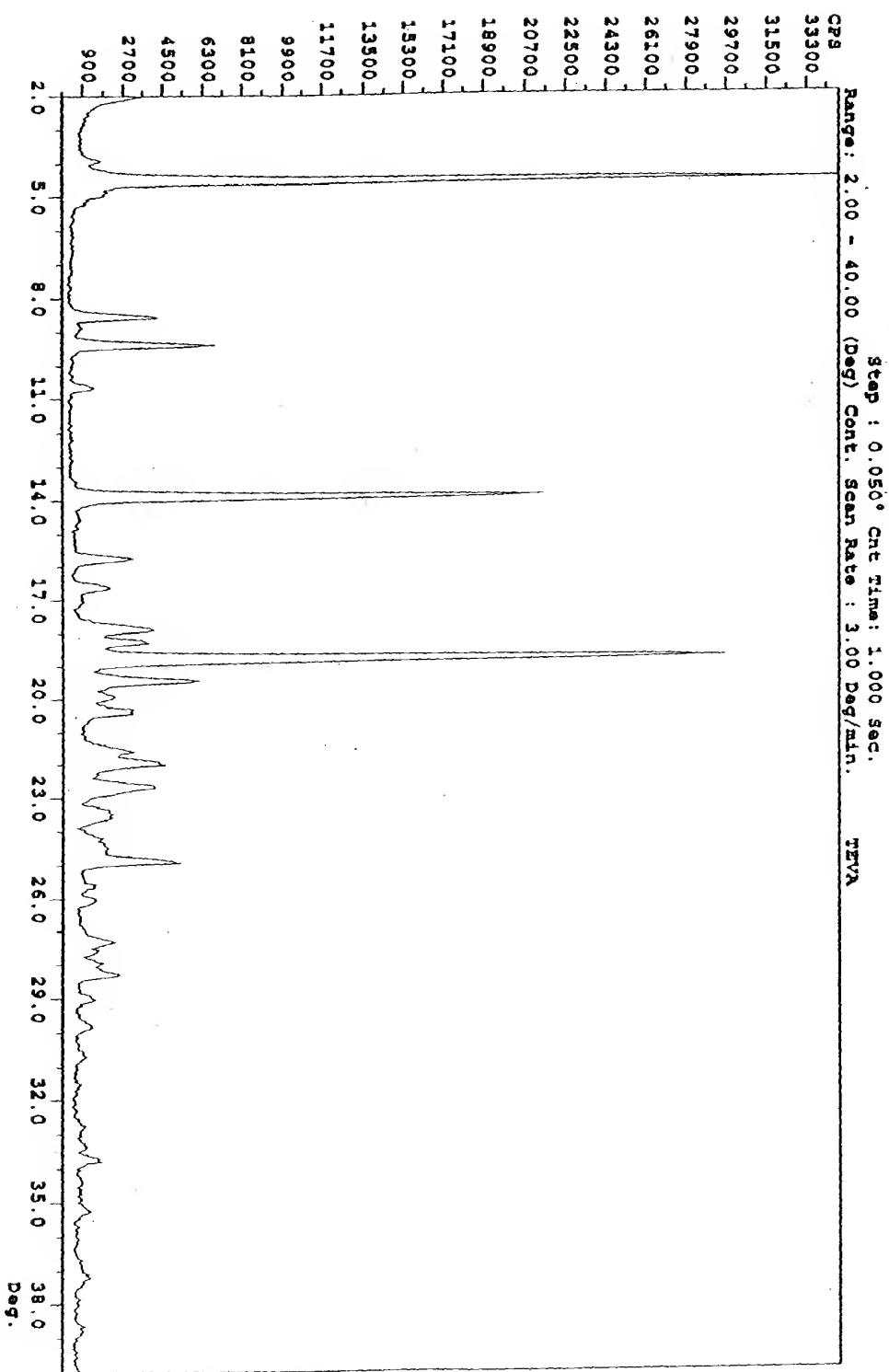


Fig 23 10

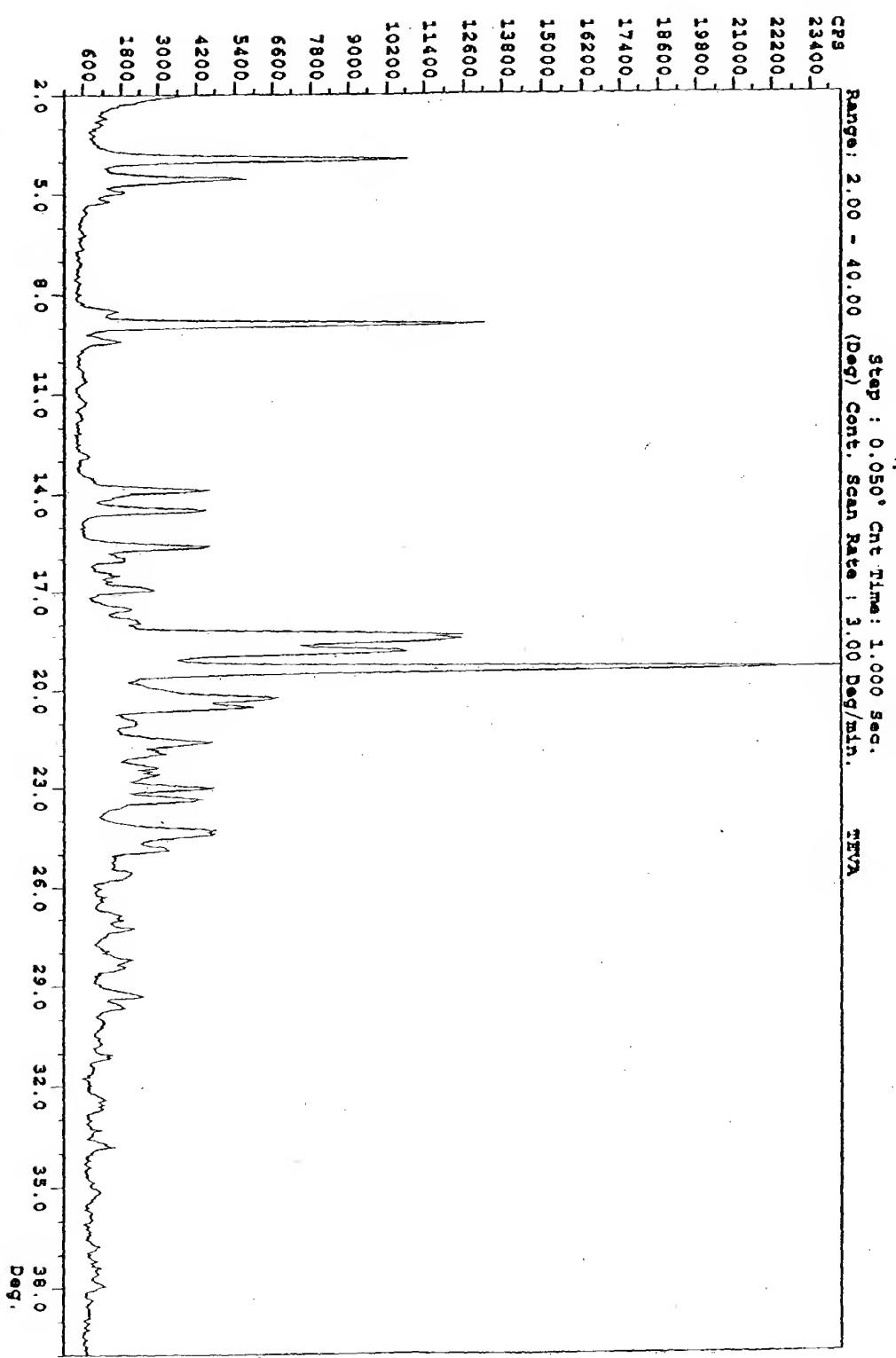


Fig 24

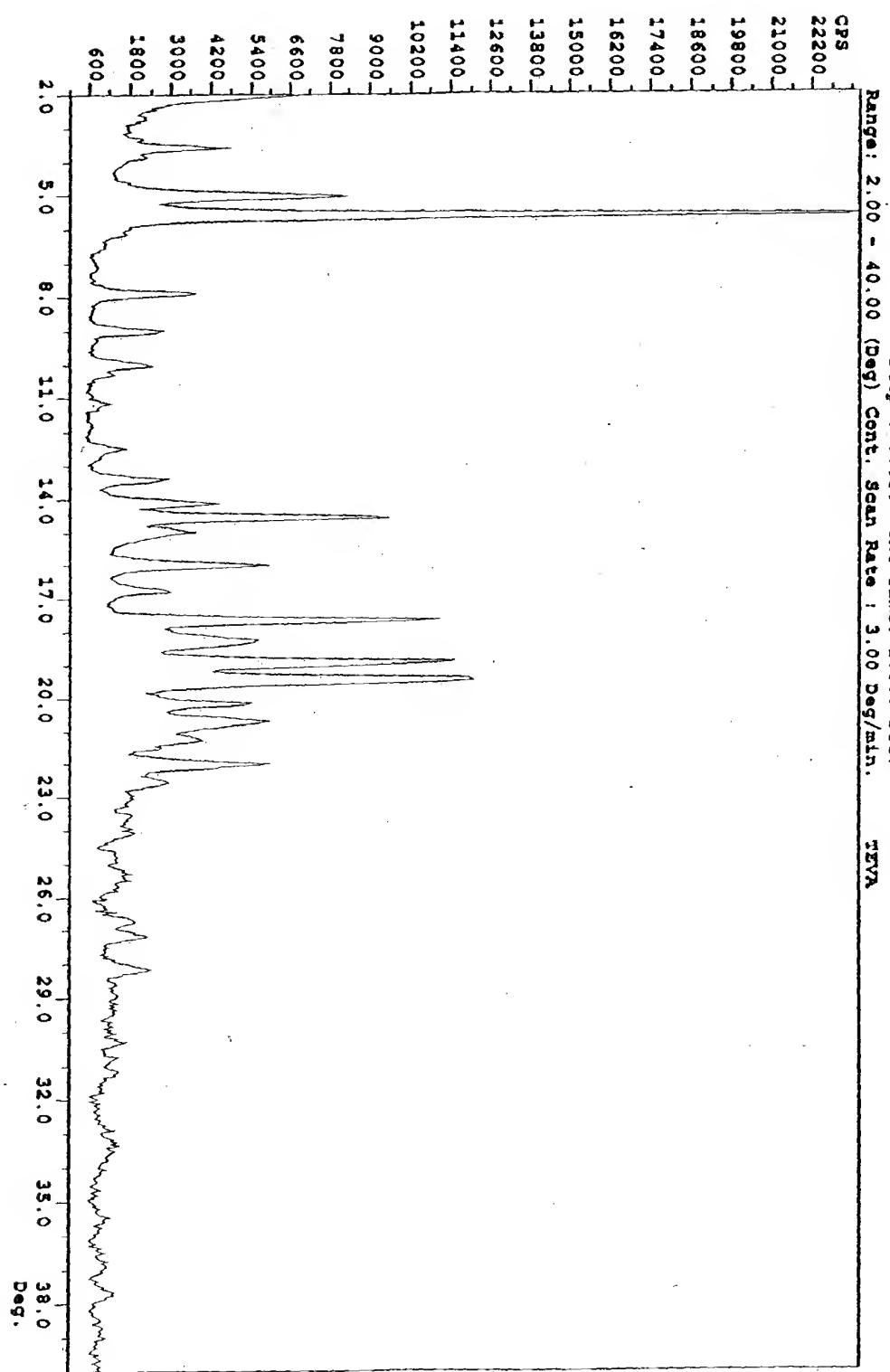


Fig 25

C

Step : 0.050° Cnt Time: 1.000 sec.
Range: 2.00 - 40.00 (Deg) Cont. scan Rate : 3.00 Deg/min. TEVA

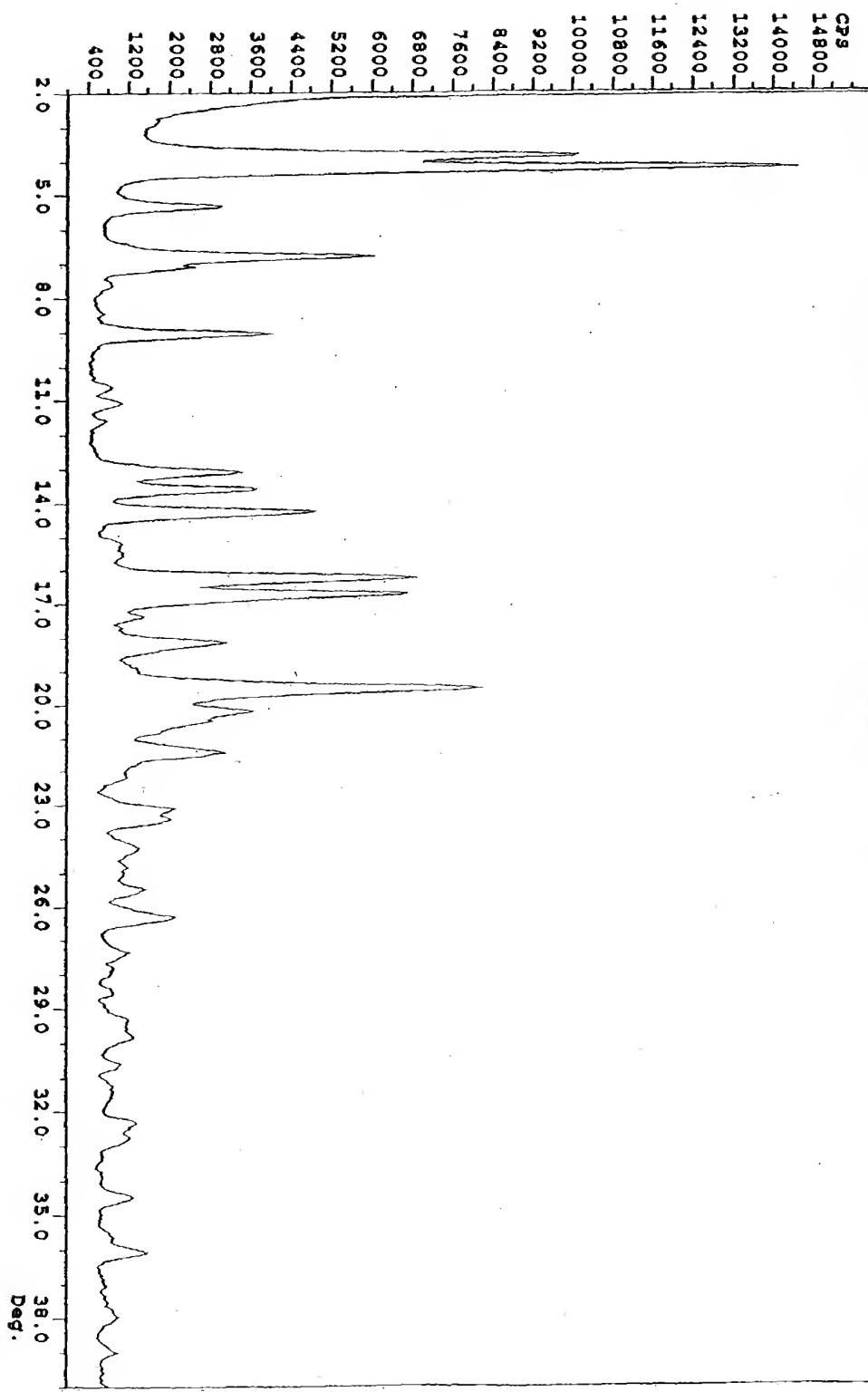


FIGURE 26

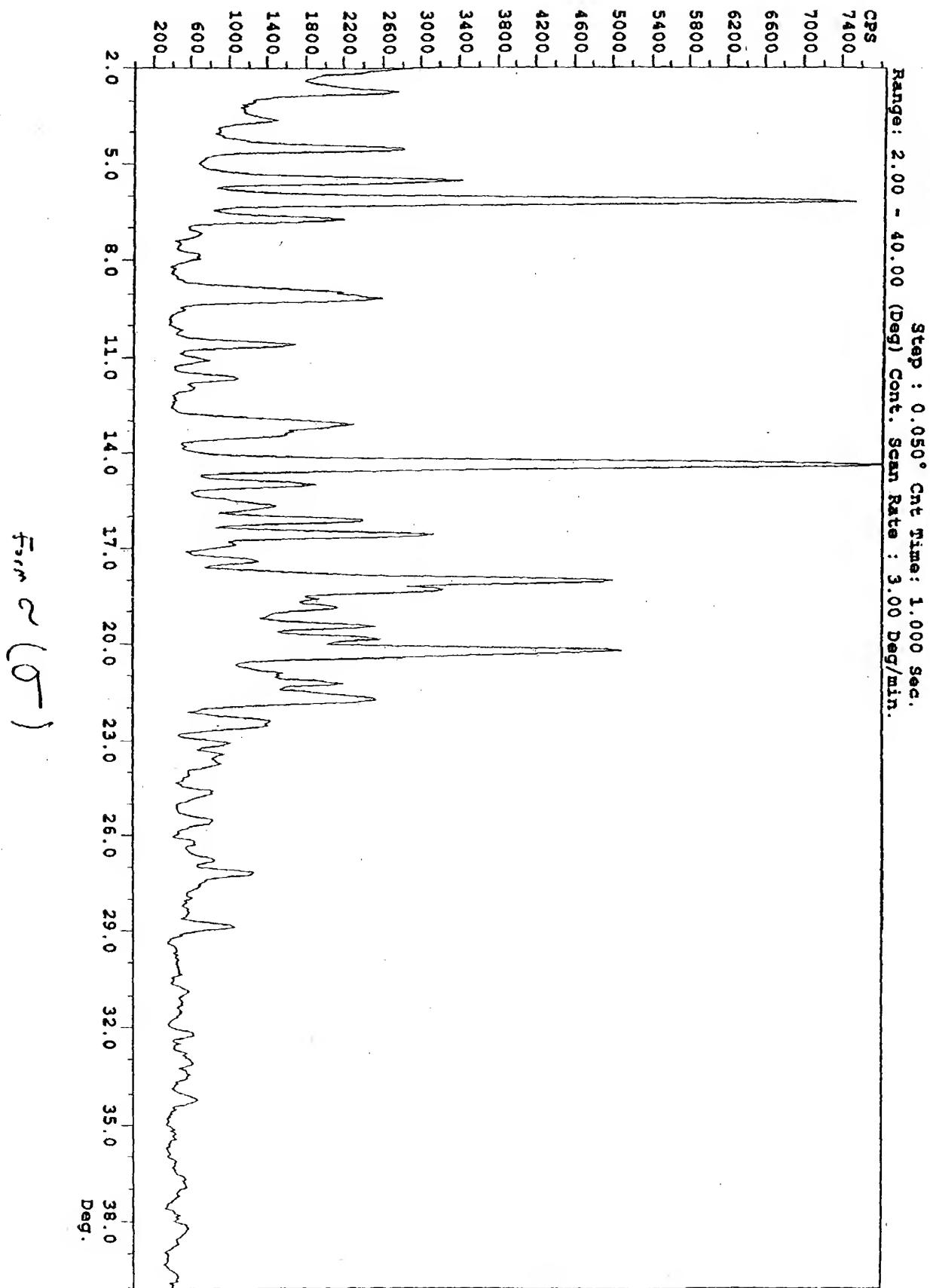
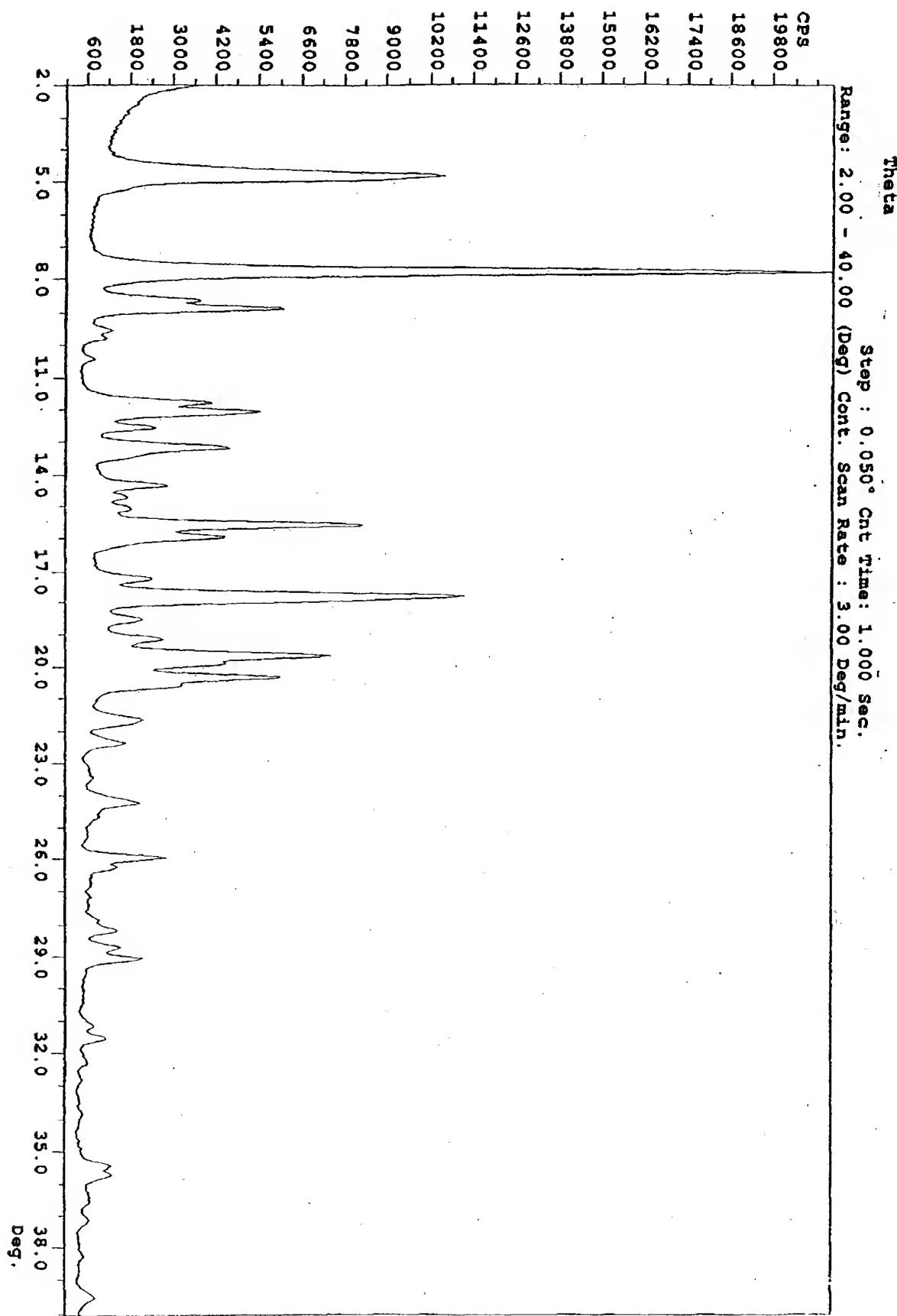


FIGURE 28.



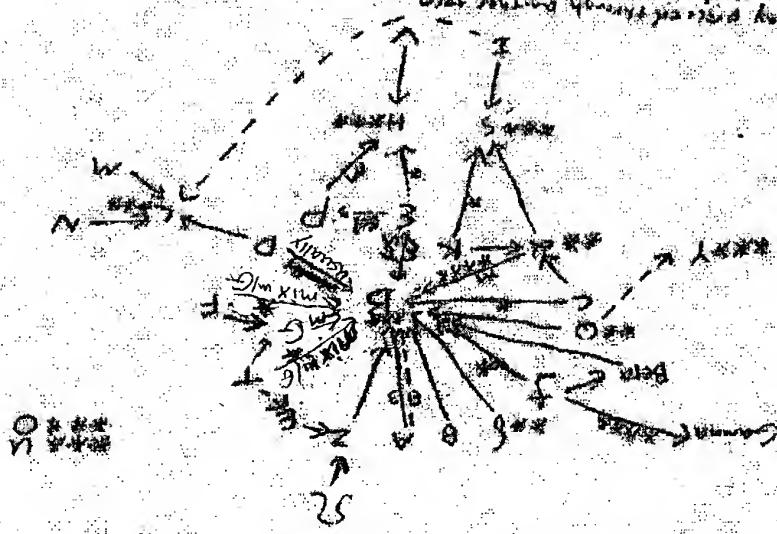
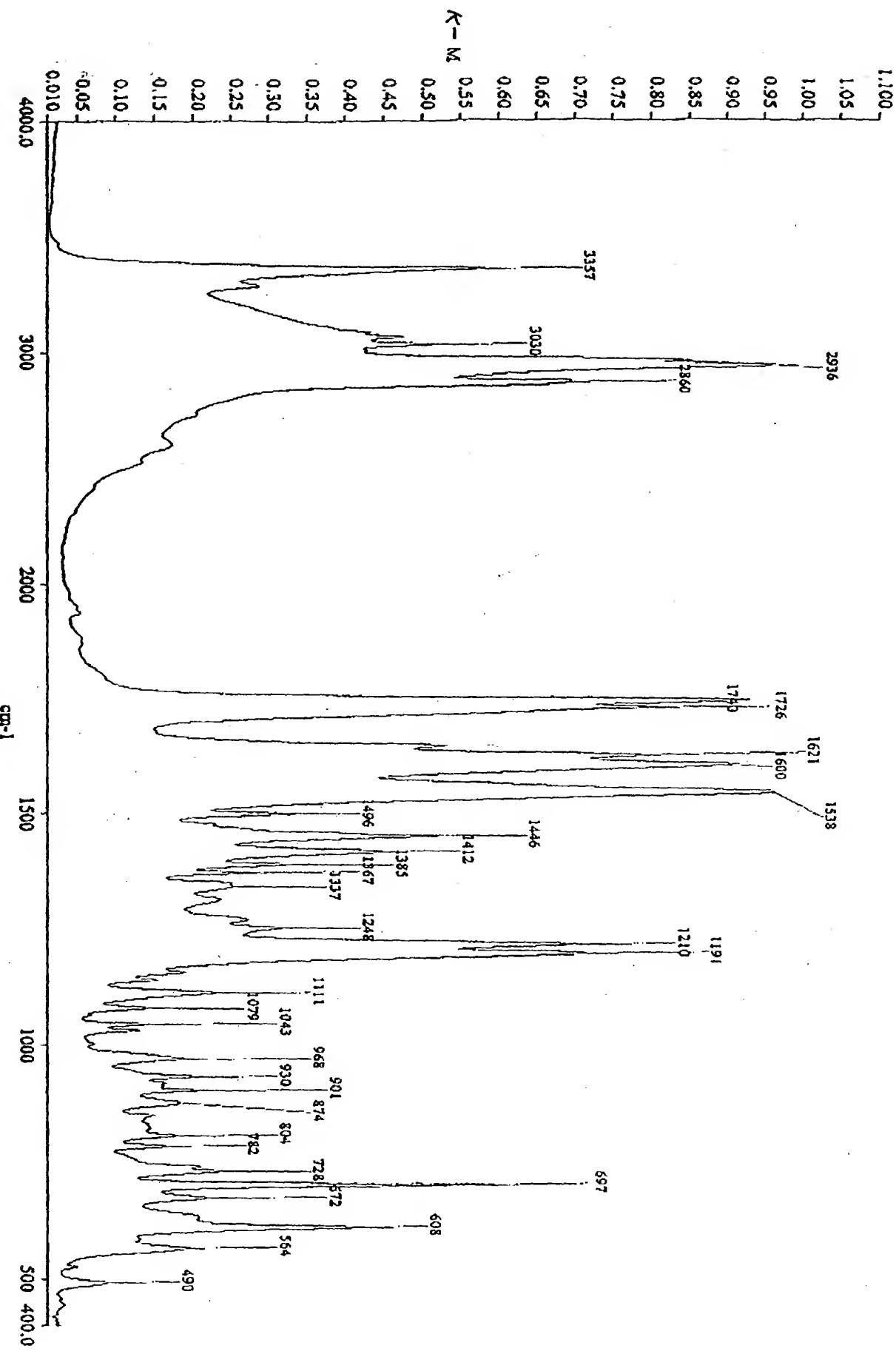


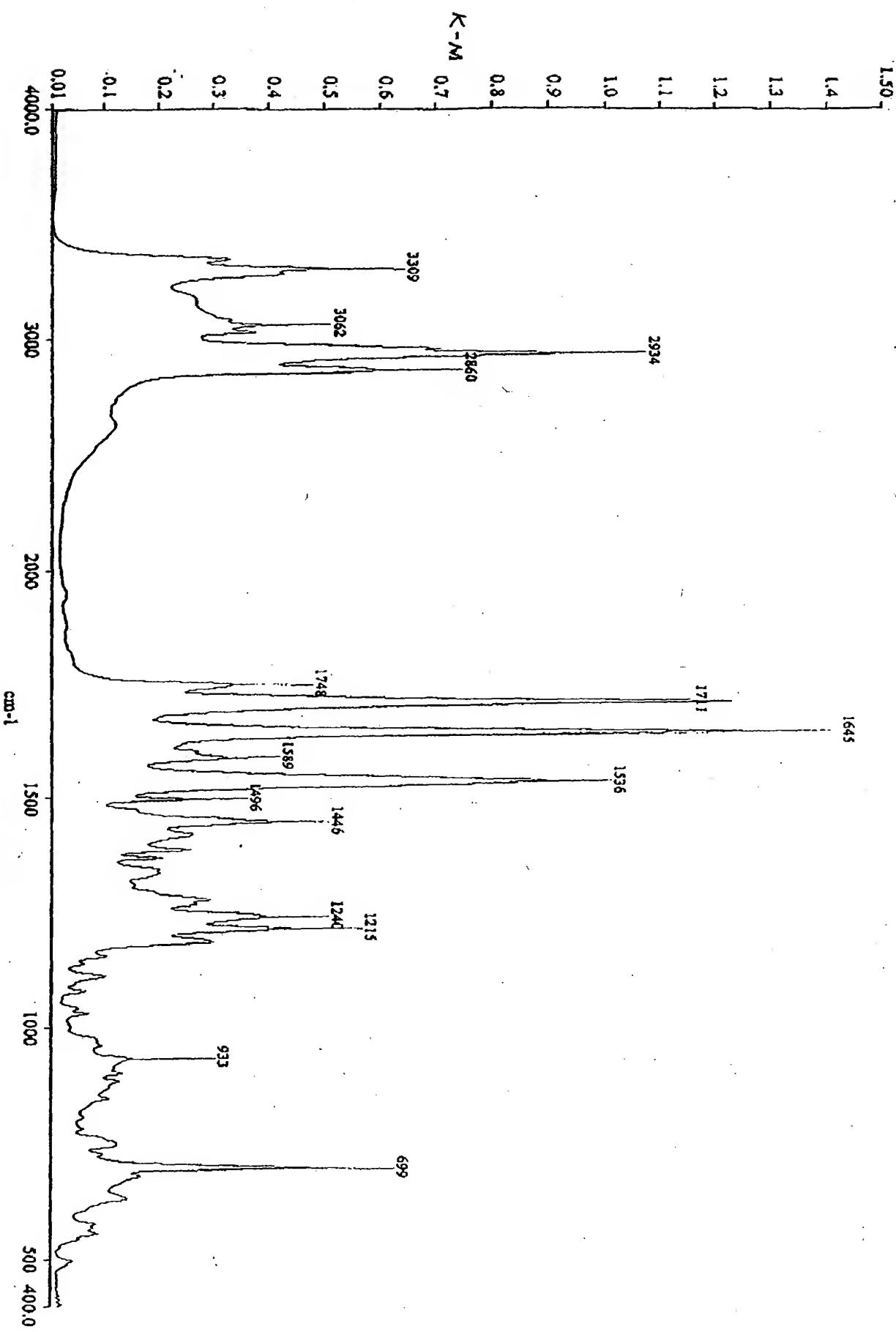
Figure 38 - Thermal stability chart

FIGURE 20
Form L



- DRIFT, 4000-400 CM⁻¹, 18 scans, Resolution: 4.00 cm⁻¹

FIGURE 30
Form P



DRIFT, 4000-400 CM⁻¹, 16 scans, Resolution: 4.00 cm⁻¹

FIGURE 5b
Form 4

-DRIFT, 4000-400cm⁻¹, 16 scans, resolution: 4.0cm⁻¹

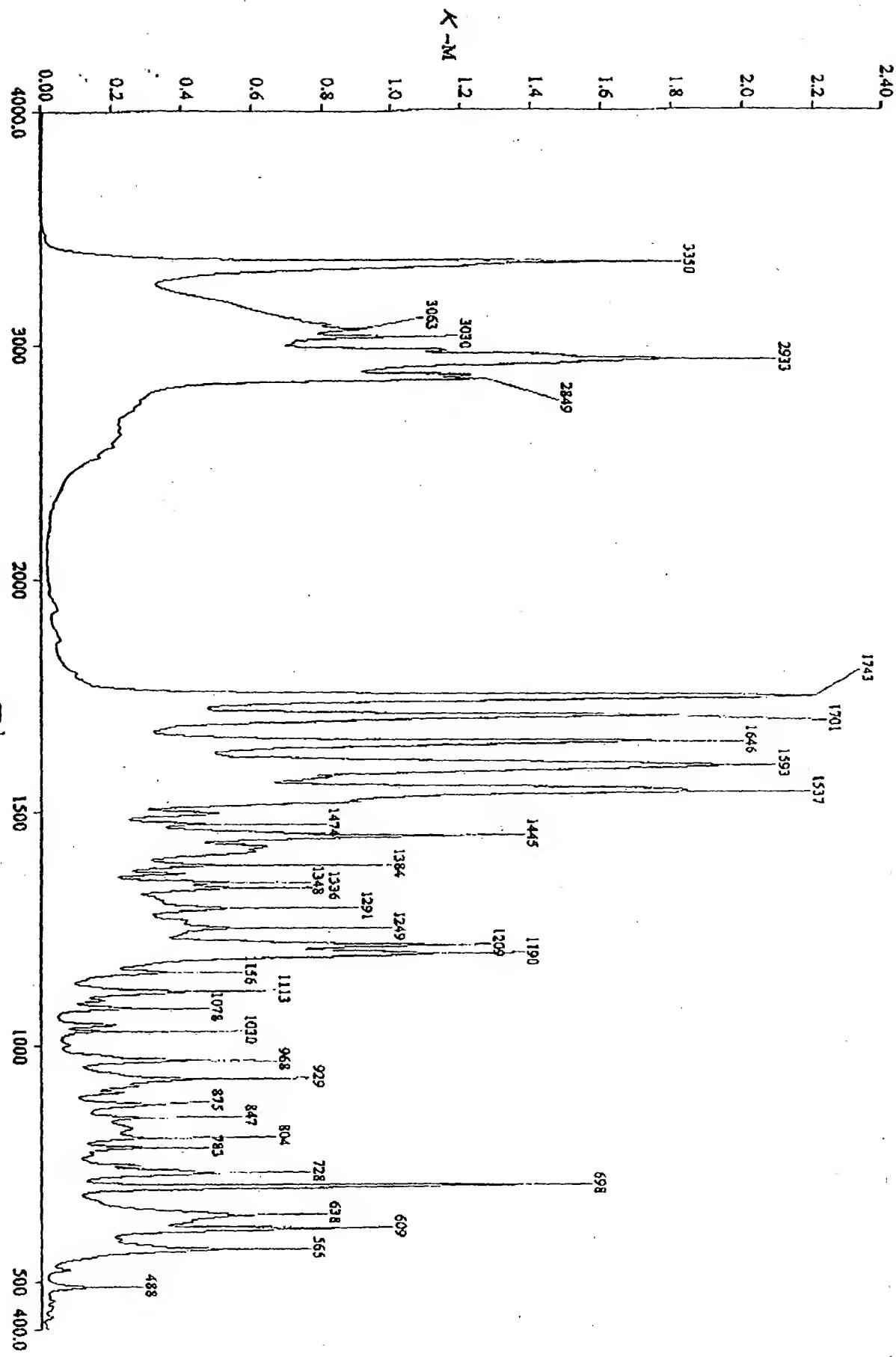


Figure 32 - Nateglinide Form 2

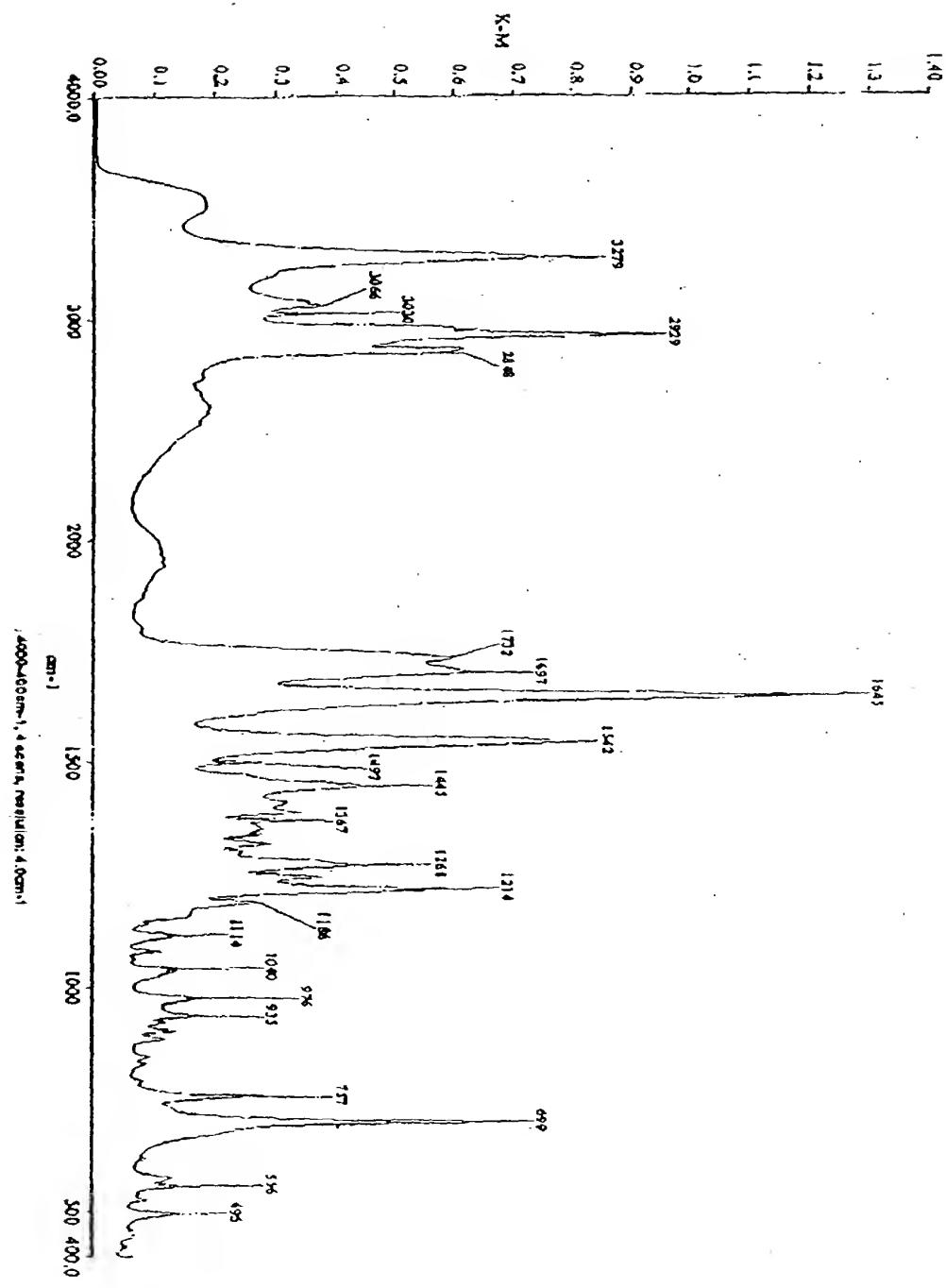


FIGURE 33
Furn

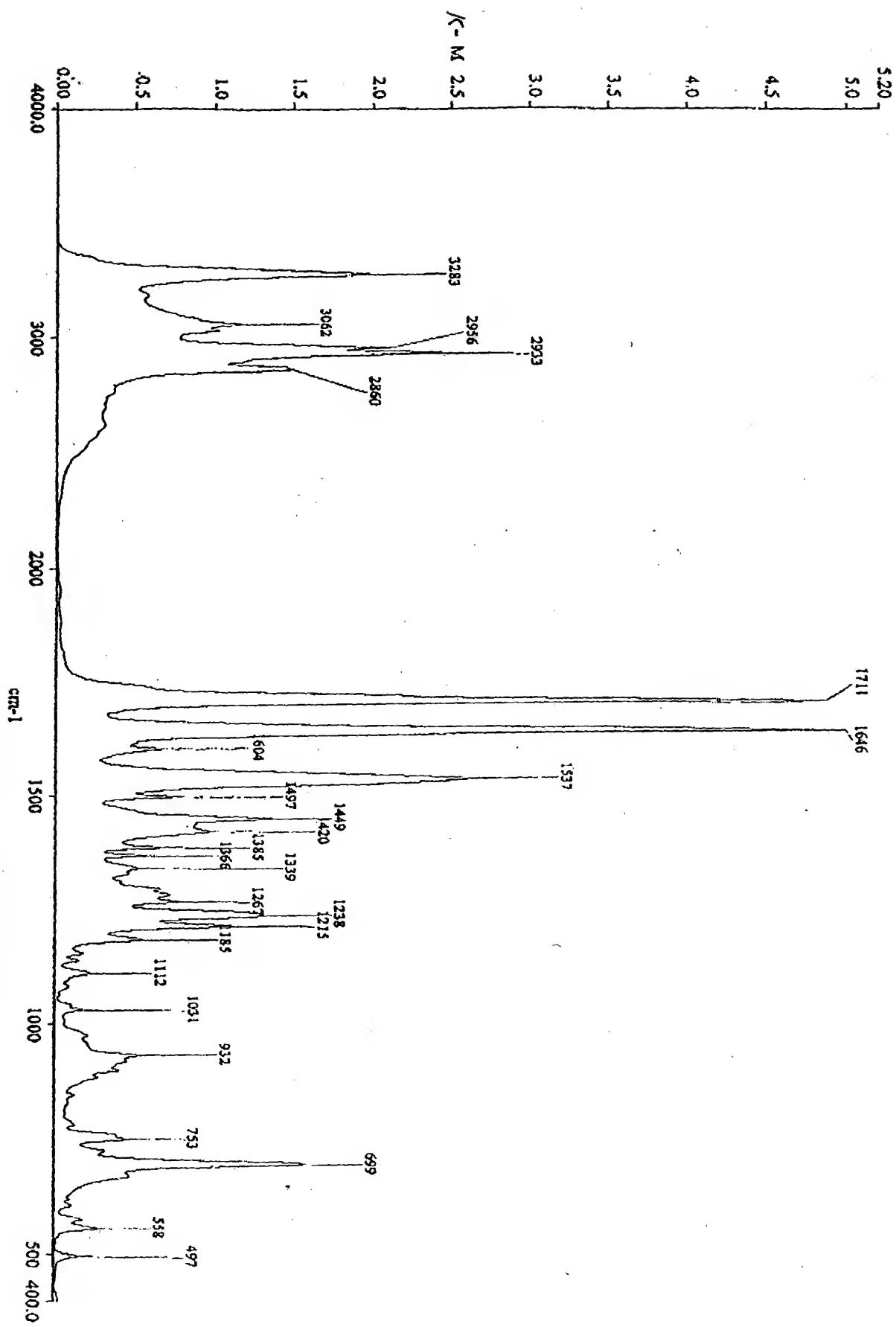
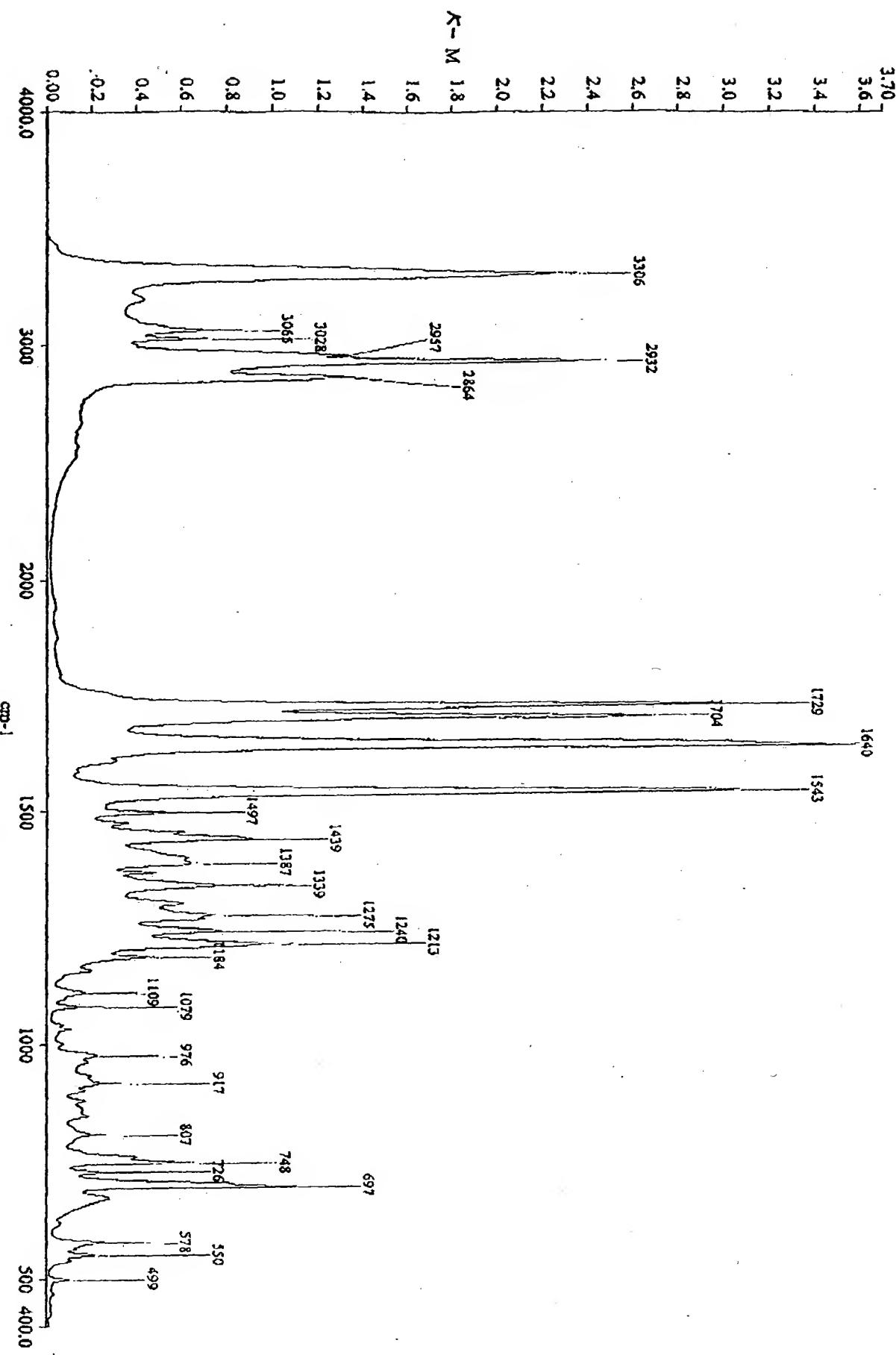


FIGURE 34
Form delta



DRIFT, 4000-400 cm^{-1} , 16 scans, resolution: 4.0 cm^{-1}

FIGURE 35 - Formic

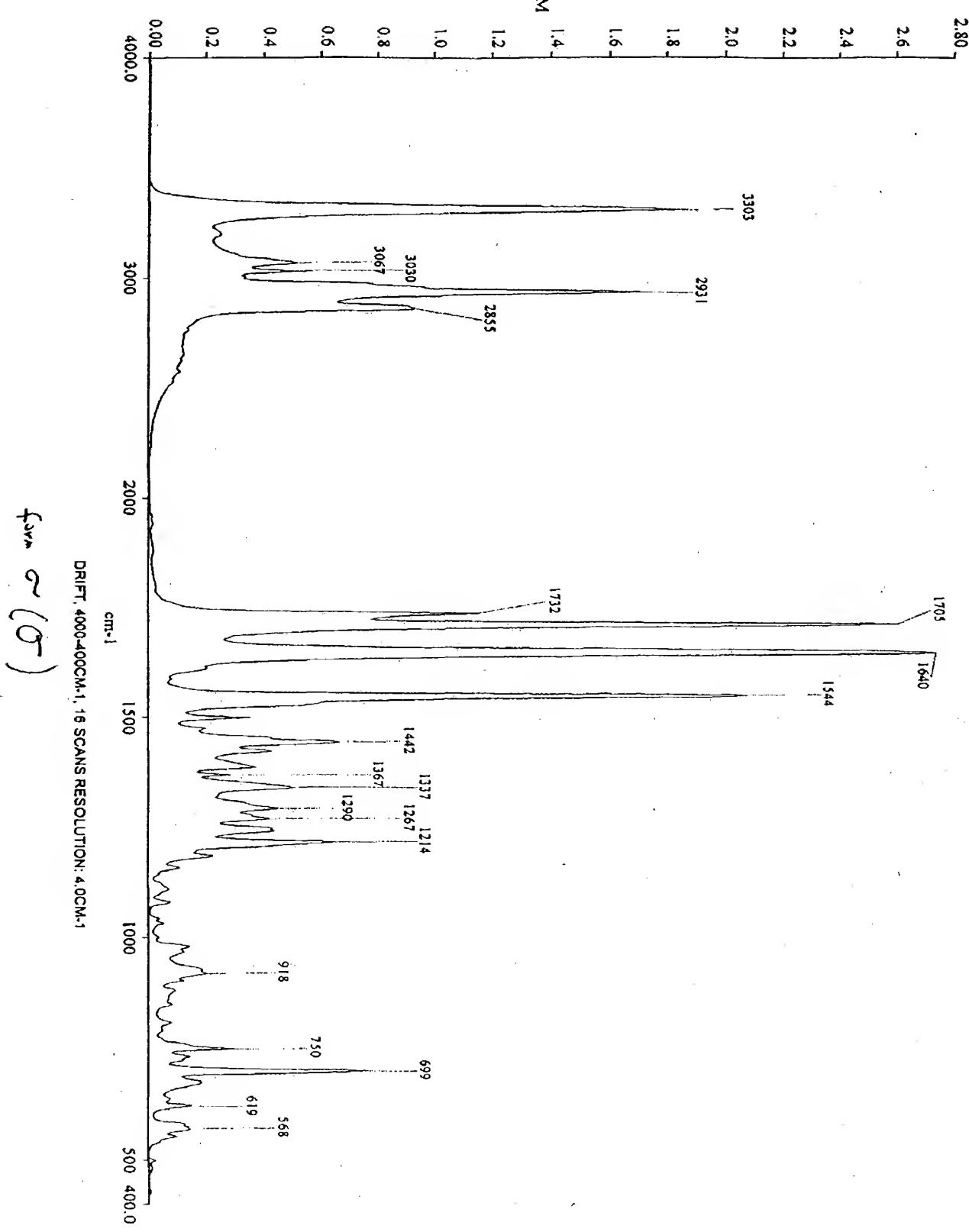


FIGURE 36
Form A

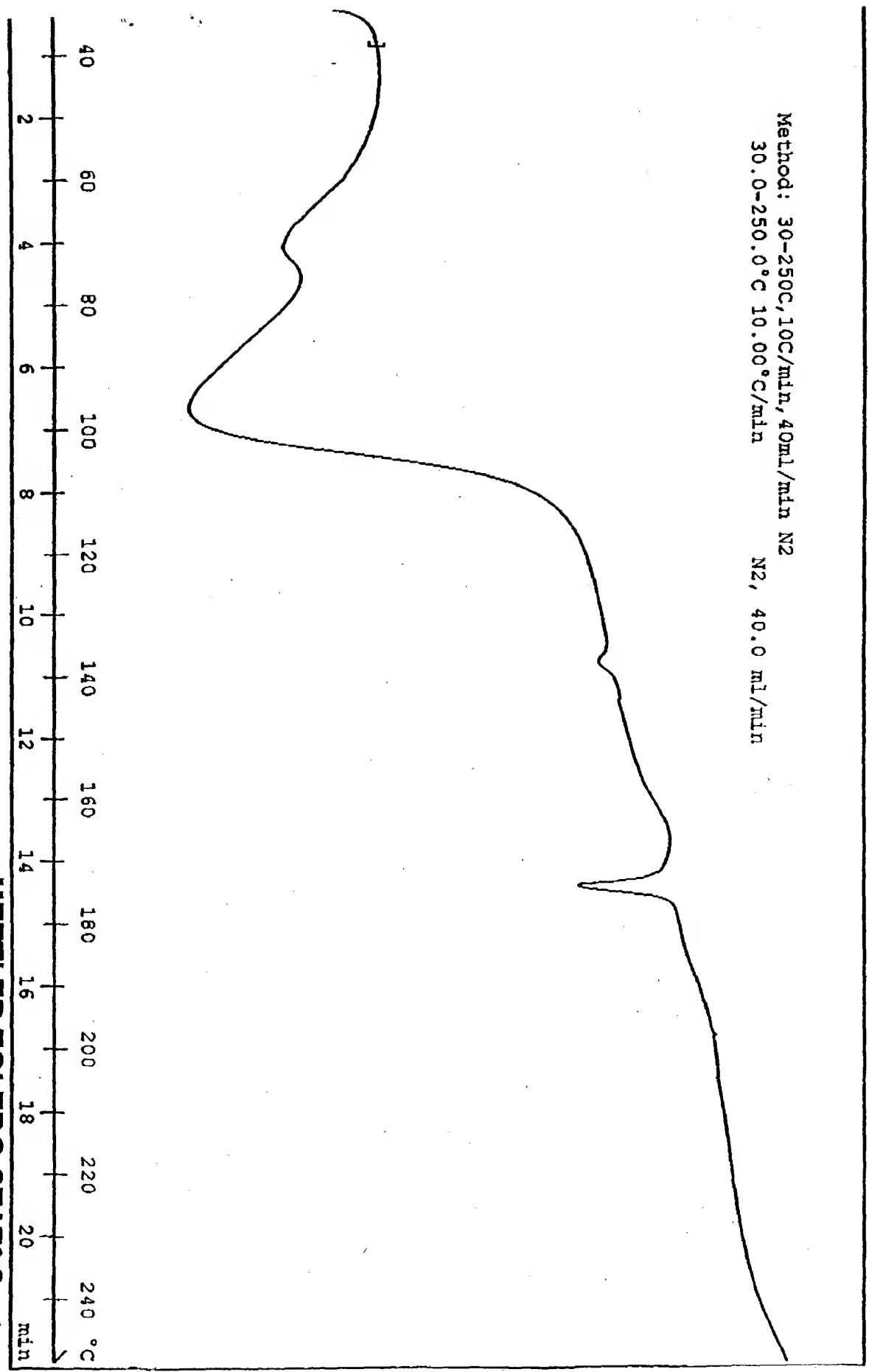
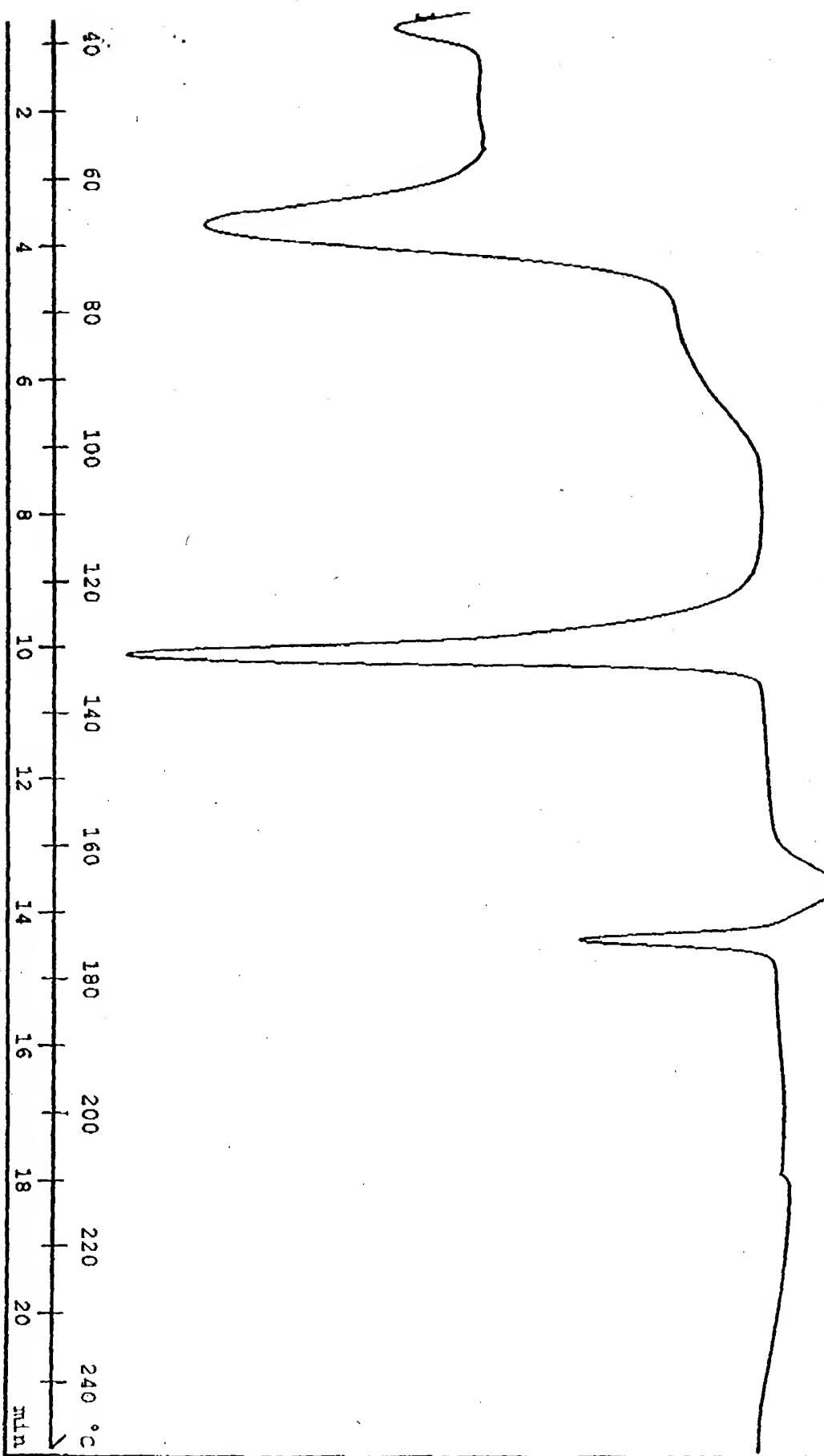


FIGURE 37 Form D

Method: 30-250.0°C, 10C/min, 40mL/min N2
30.0-250.0°C 10.00°C/min
N2, 40.0 mL/min

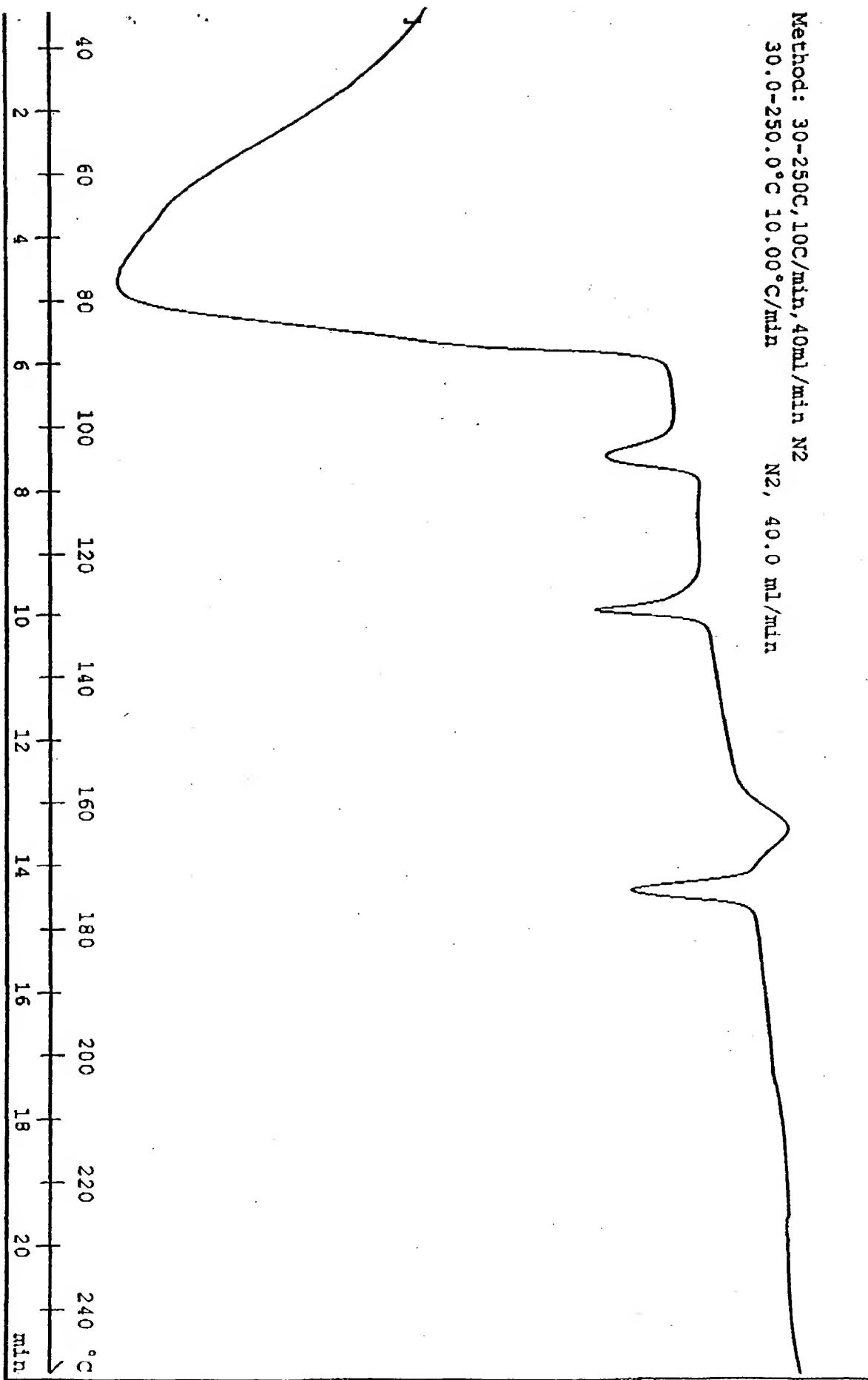


METTLER TOLEDO STAR® System

FIGURE 38

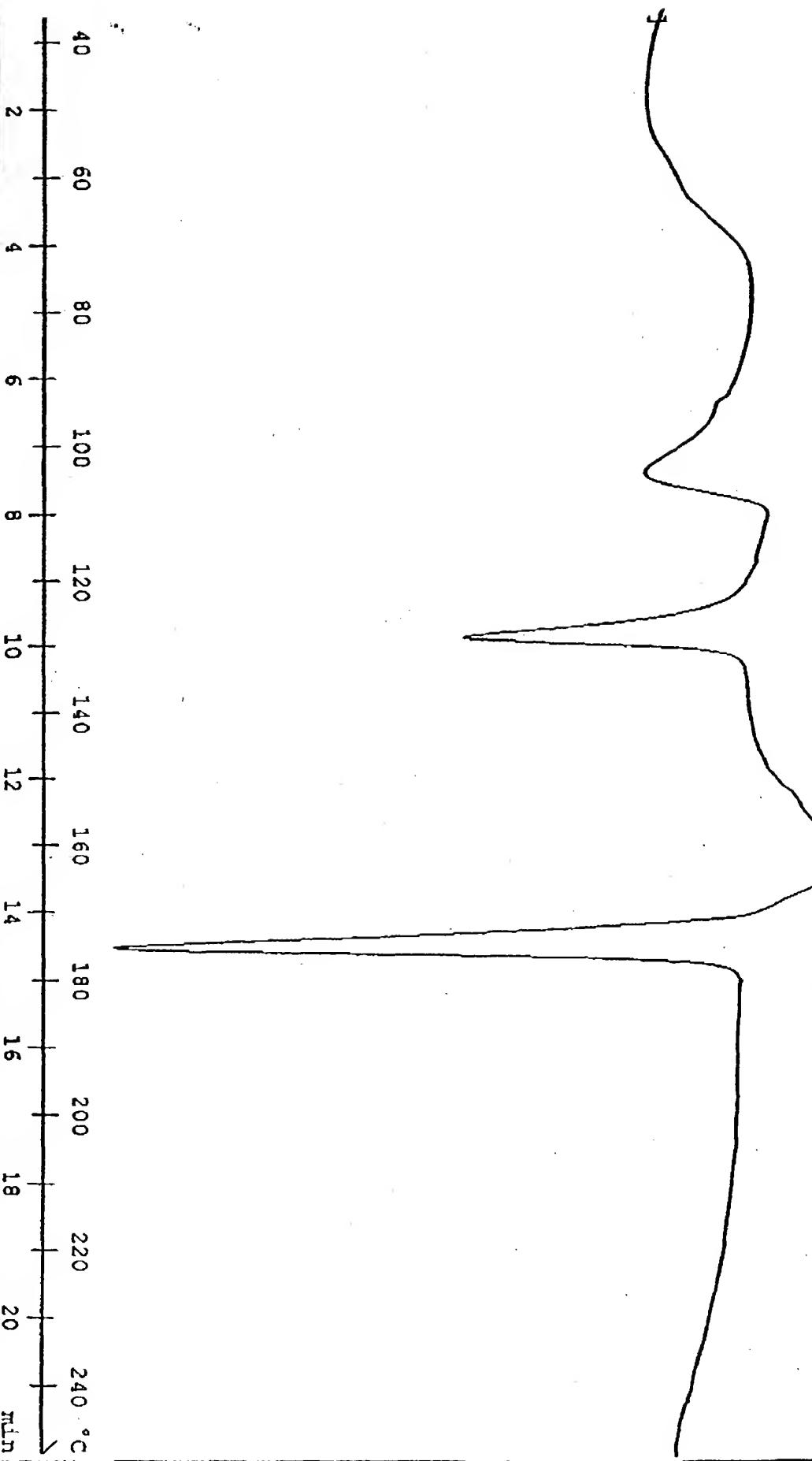
Form E

Method: 30-250C, 10C/min, 40ml/min N2
30.0-250.0°C 10.00°C/min N2, 40.0 ml/min



39
FIGURE

Method: 30-250.0°C, 10C/min, 40mL/min N2
30.0-250.0°C 10.00°C/min
N2, 40.0 mL/min

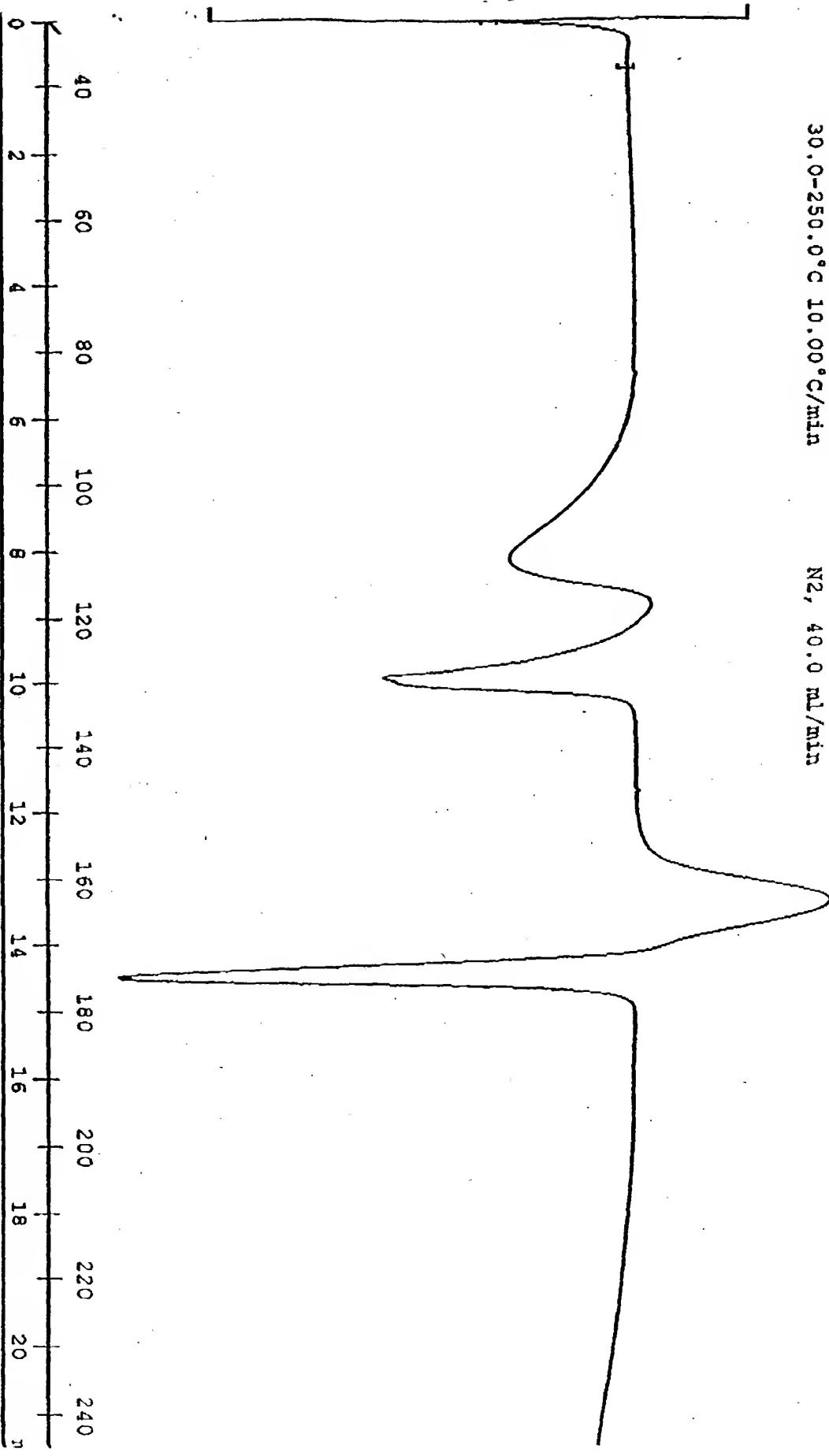


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40
FIGURE 28

1X0

Method: 30-250C, 10C/min, 40ml/min N2
30.0-250.0°C 10.00°C/min N2, 40.0 ml/min



METTLER TOLEDO STAR System

FIGURE 41
Form I

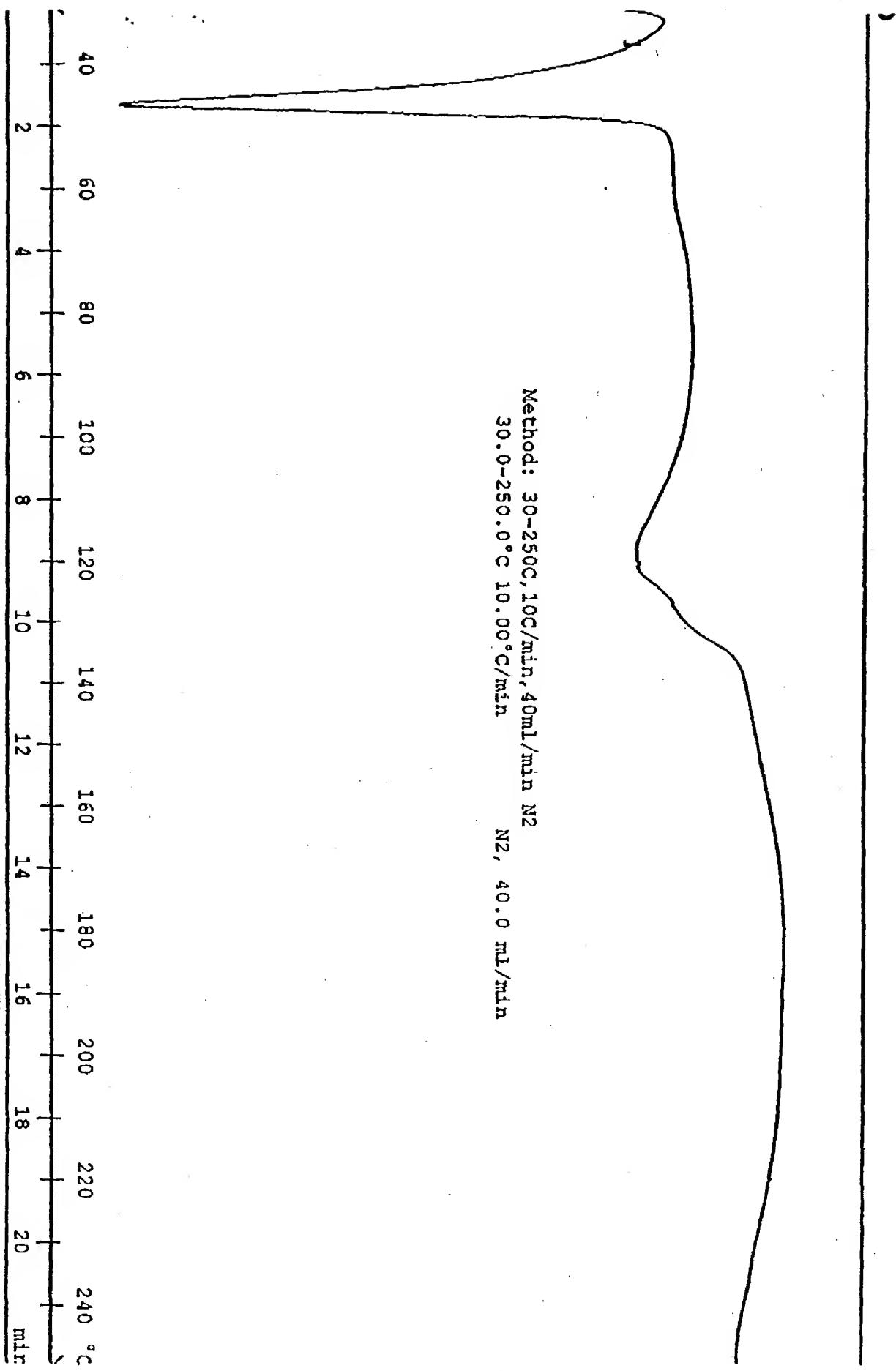


FIGURE 49
Form T

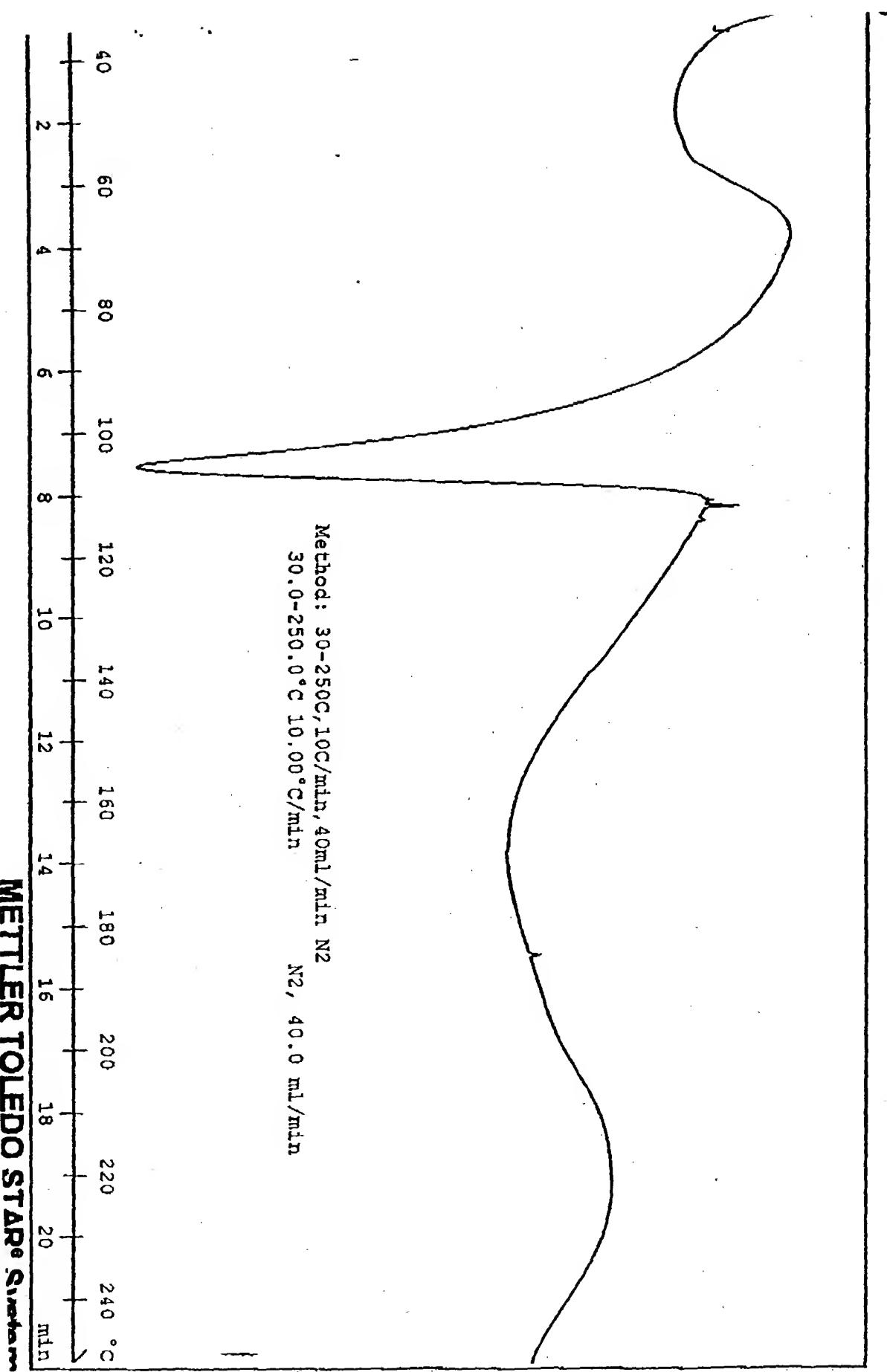


FIGURE 43
Form K

Method: 30-250C, 10C/min, 40ml/min N2
30.0-250.0°C 10.00°C/min. N2, 40.0 ml/min

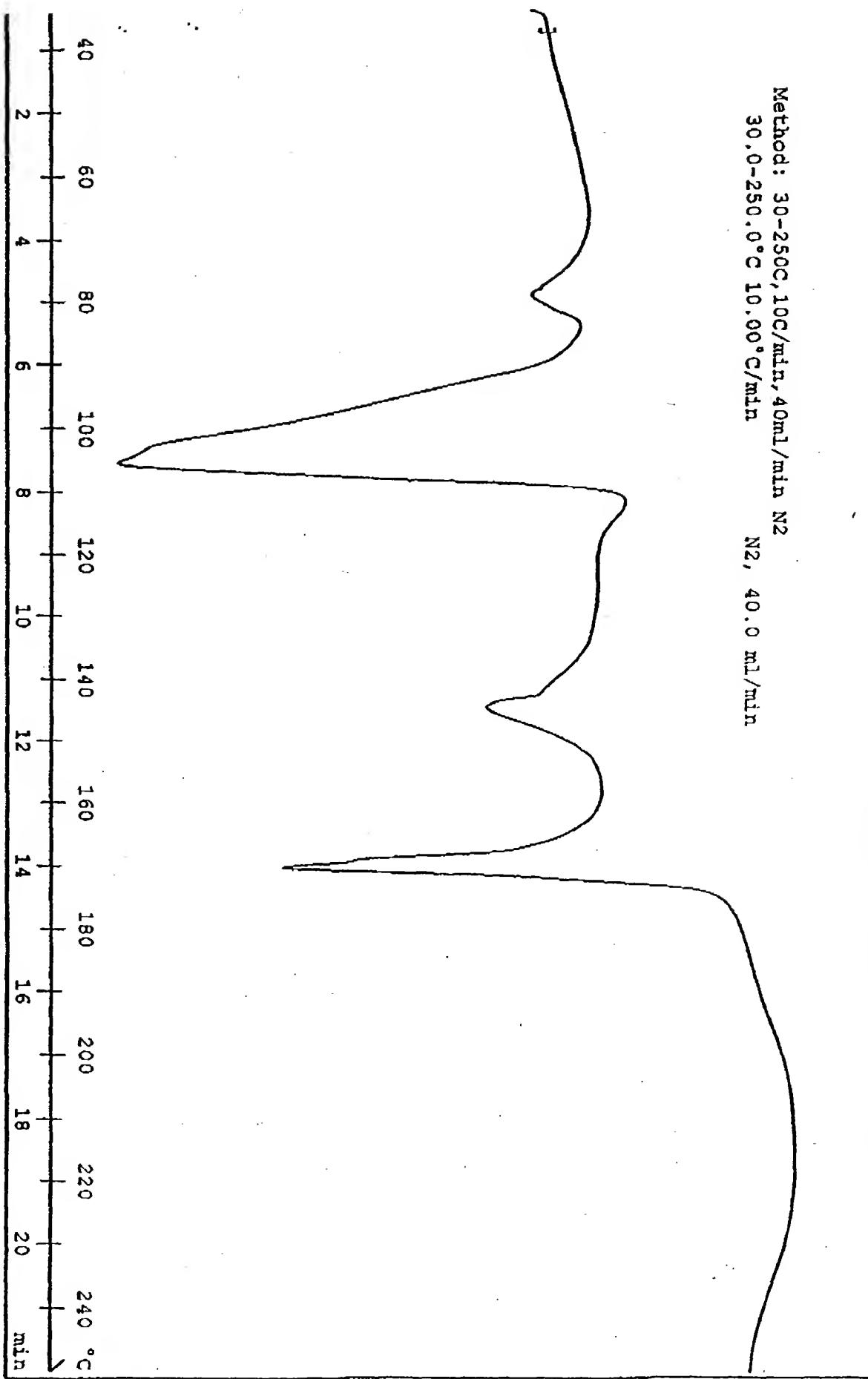
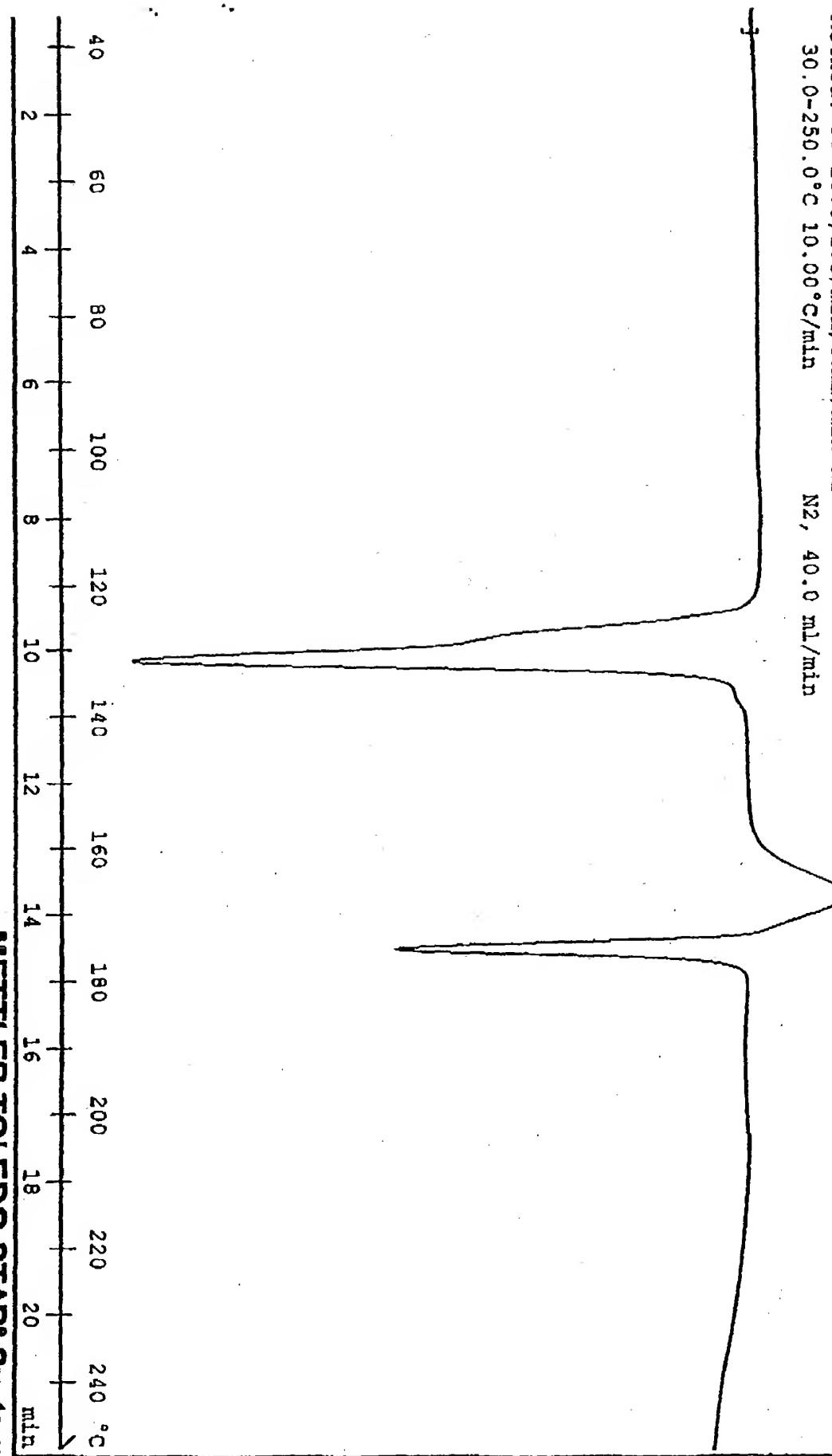


FIGURE 42 4/4
Form L

Method: 30-250°C, 10C/min; 40ml/min N₂
30.0-250.0°C 10.00°C/min
N₂, 40.0 mL/min



METTLER TOI FN STAB. 0.14

Figure 45
Form M

Method: 30-250°C, 10°C/min, 40mL/min N2
30.0-250.0 °C 10.00 °C/min
N2, 40.0 mL/min

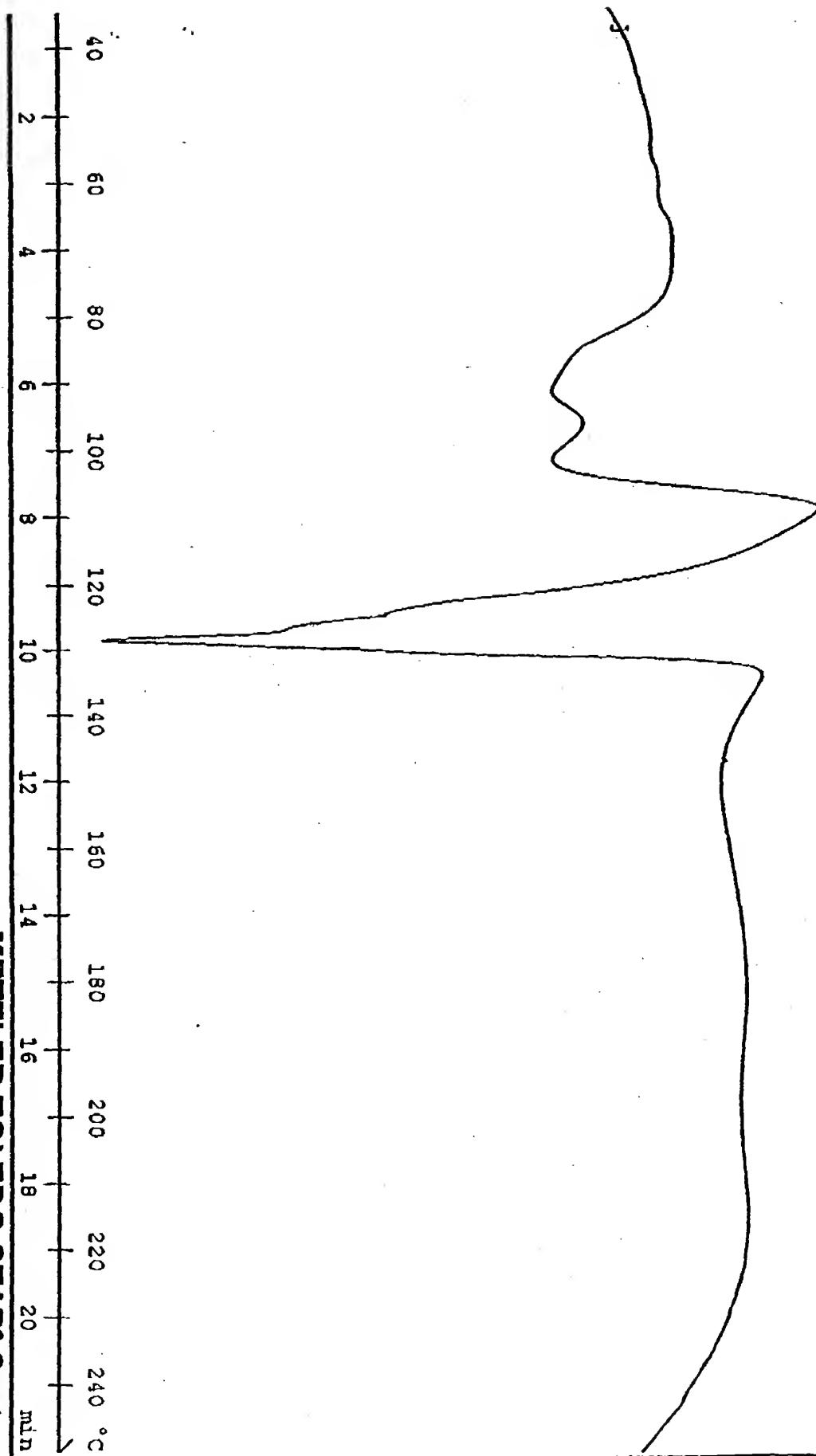
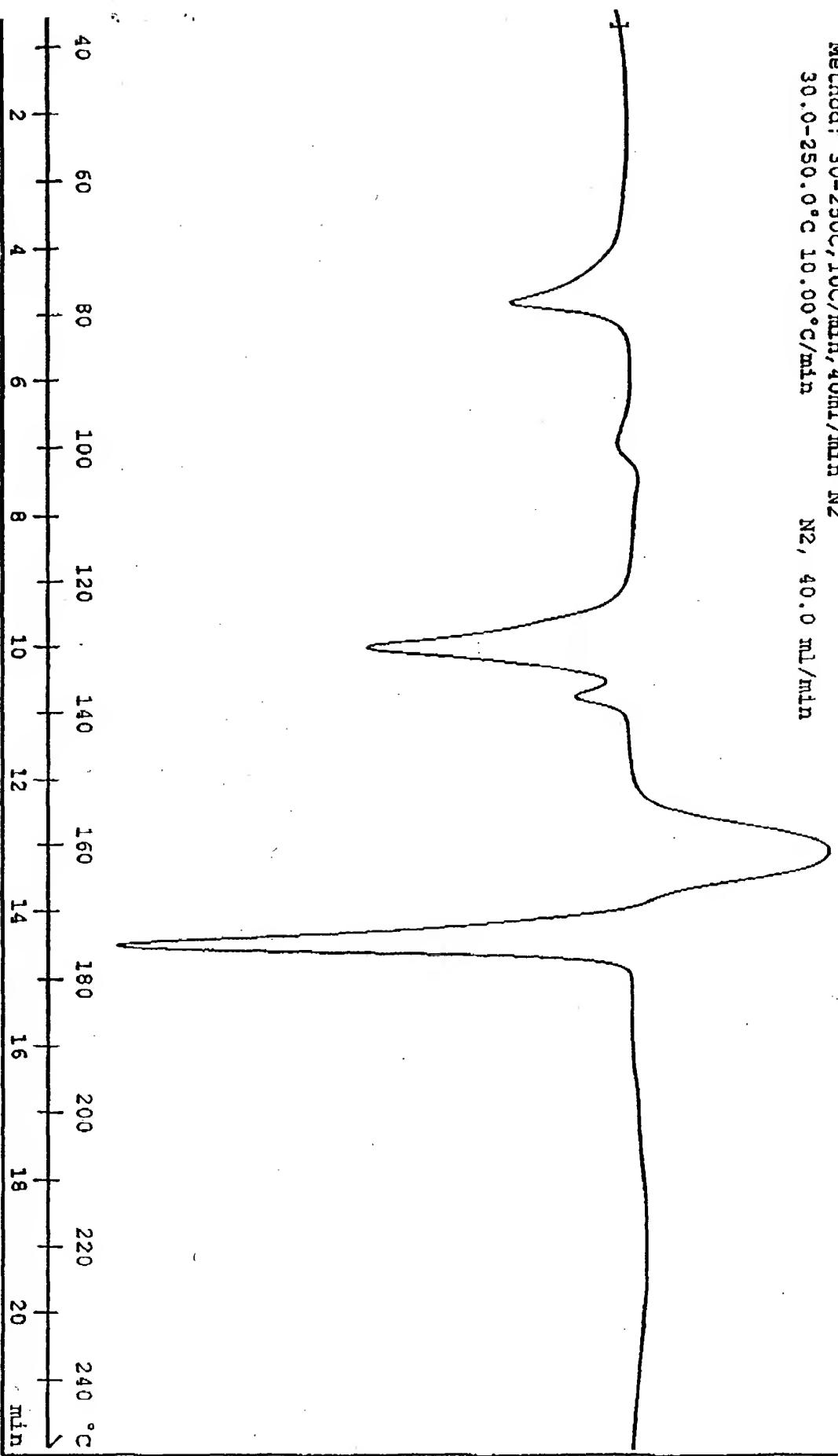


FIGURE 46
Form N

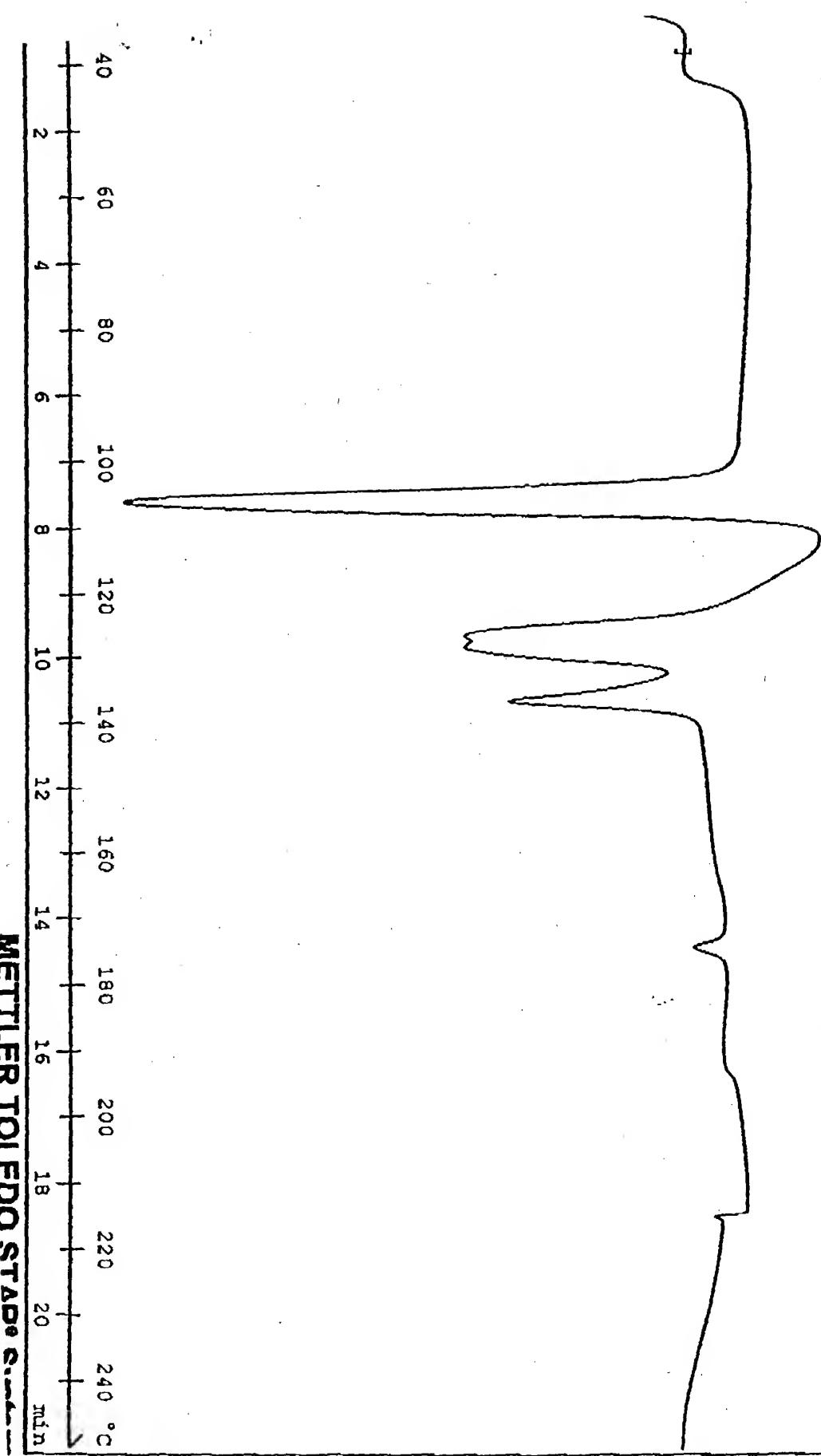
Method: 30-250°C, 10C/min, 40mL/min N₂
30.0-250.0°C 10.00°C/min N₂, 40.0 mL/min



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FIGURE 45 47
Form C

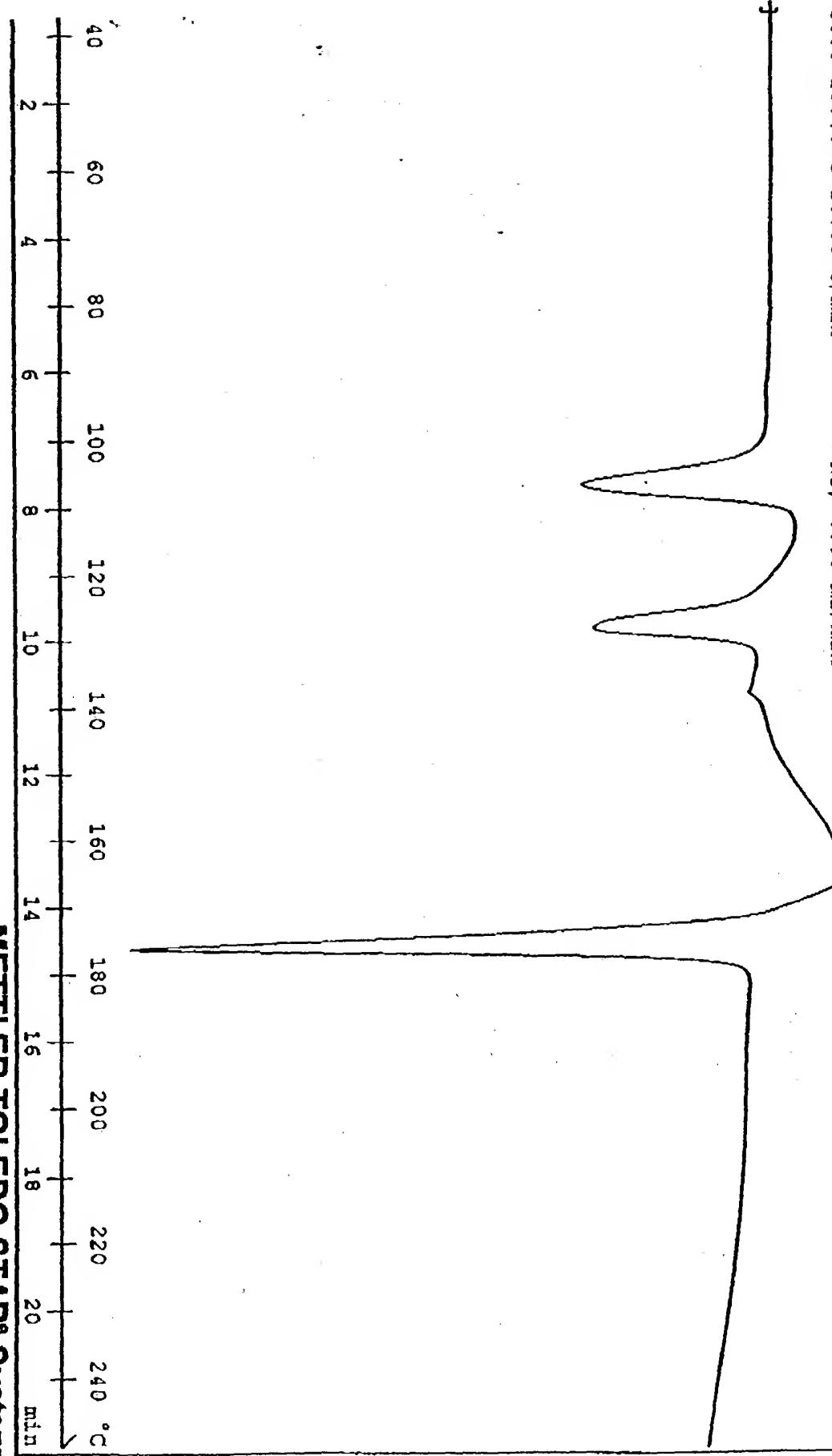
Method: 30-250C, 10C/min, 40ml/min N2
30.0-250.0°C 10.00°C/min
N2, 40.0 ml/min



METTLER TOLEDO STAB®

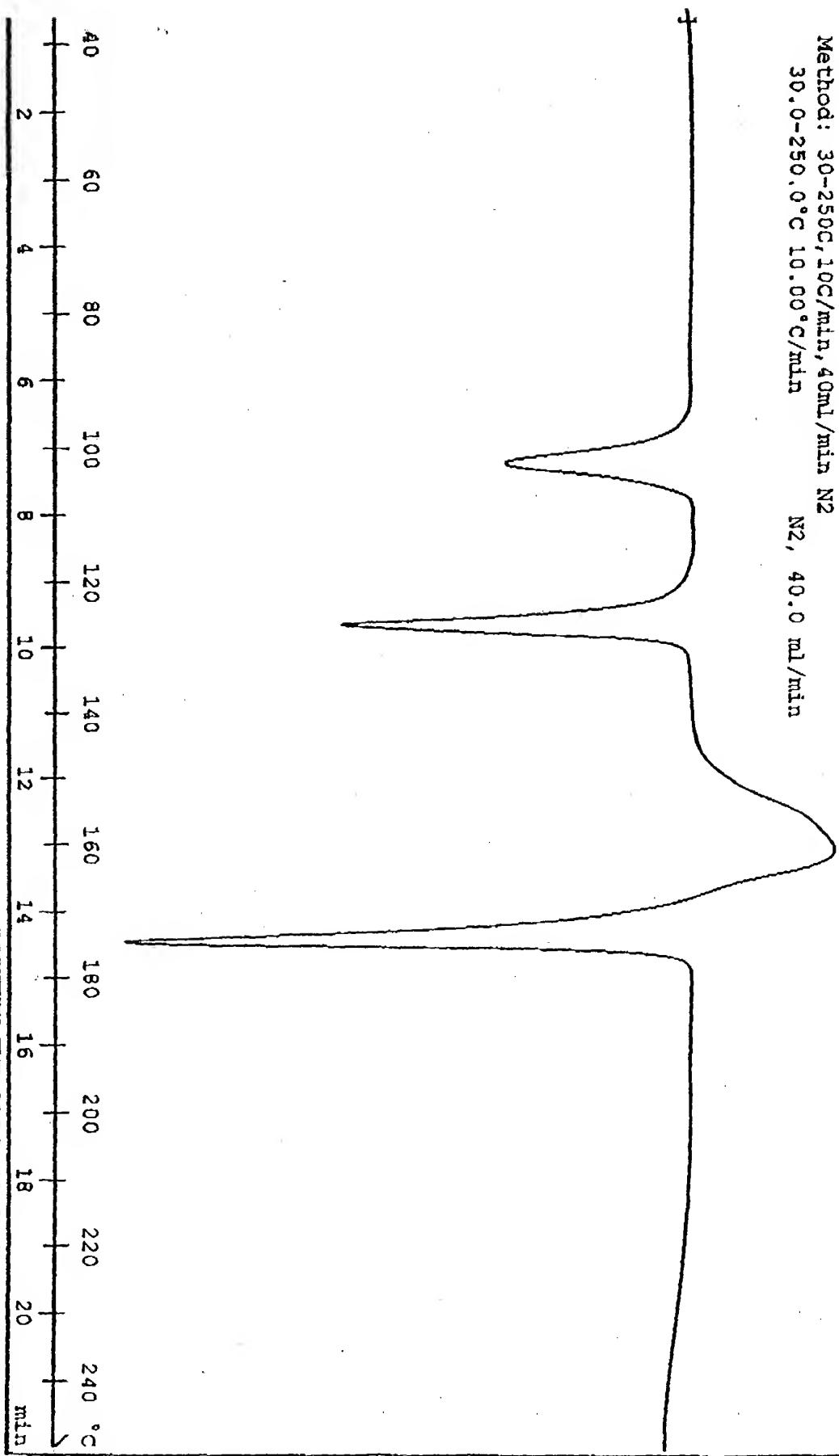
FIGURE 48
Form P

Method: 30-250C, 10C/min, 40mL/min N2
30.0-250.0°C 10.00°C/min N2, 40.0 mL/min



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FIGURE 47 49
Form C



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FIGURE 48 50
Form T

Method: 30-250C, 10C/min, 40mL/min N₂
30.0-250.0°C 10.00°C/min N₂, 40.0 mL/min

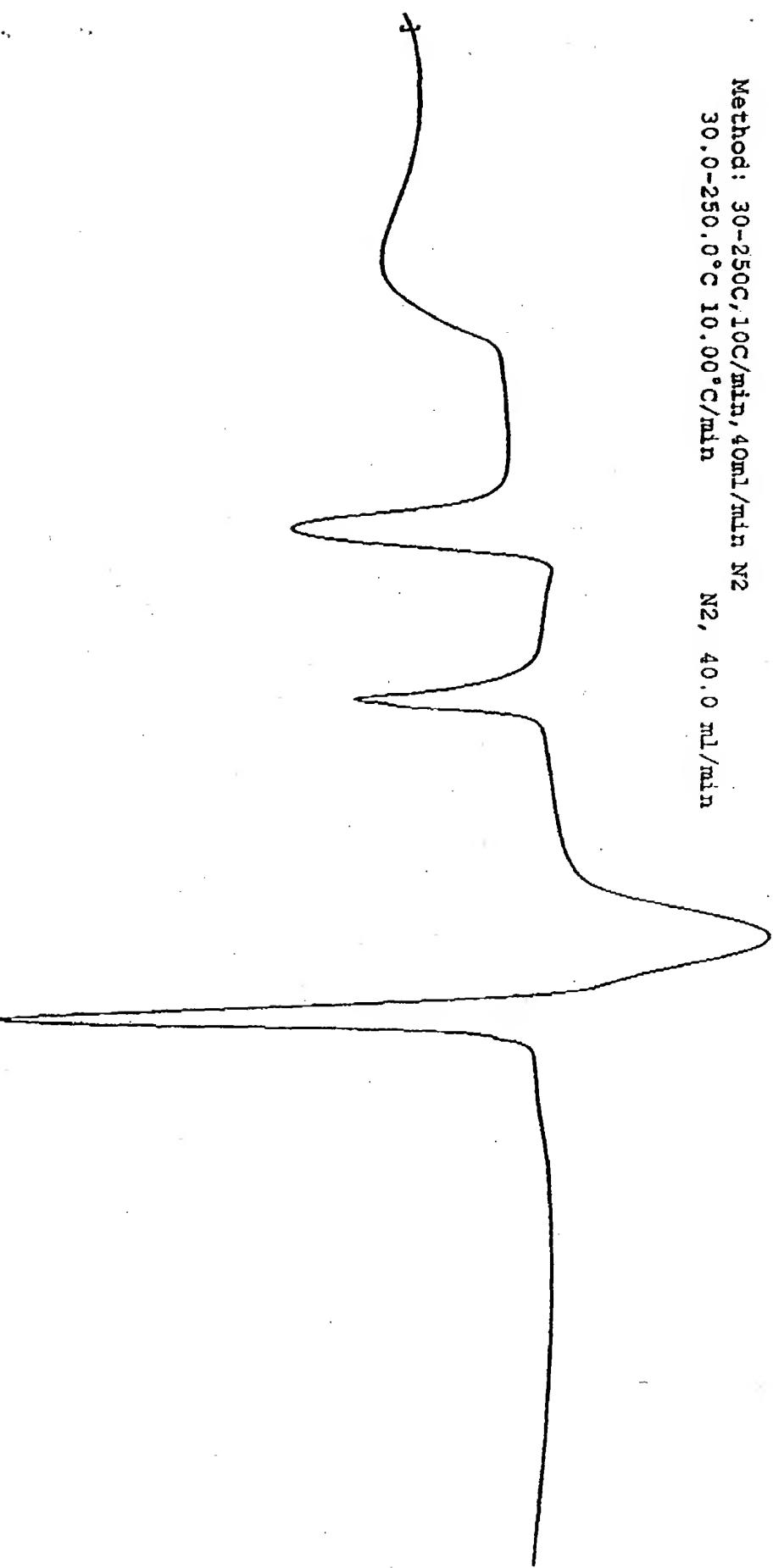


FIGURE 49. 51
Form W

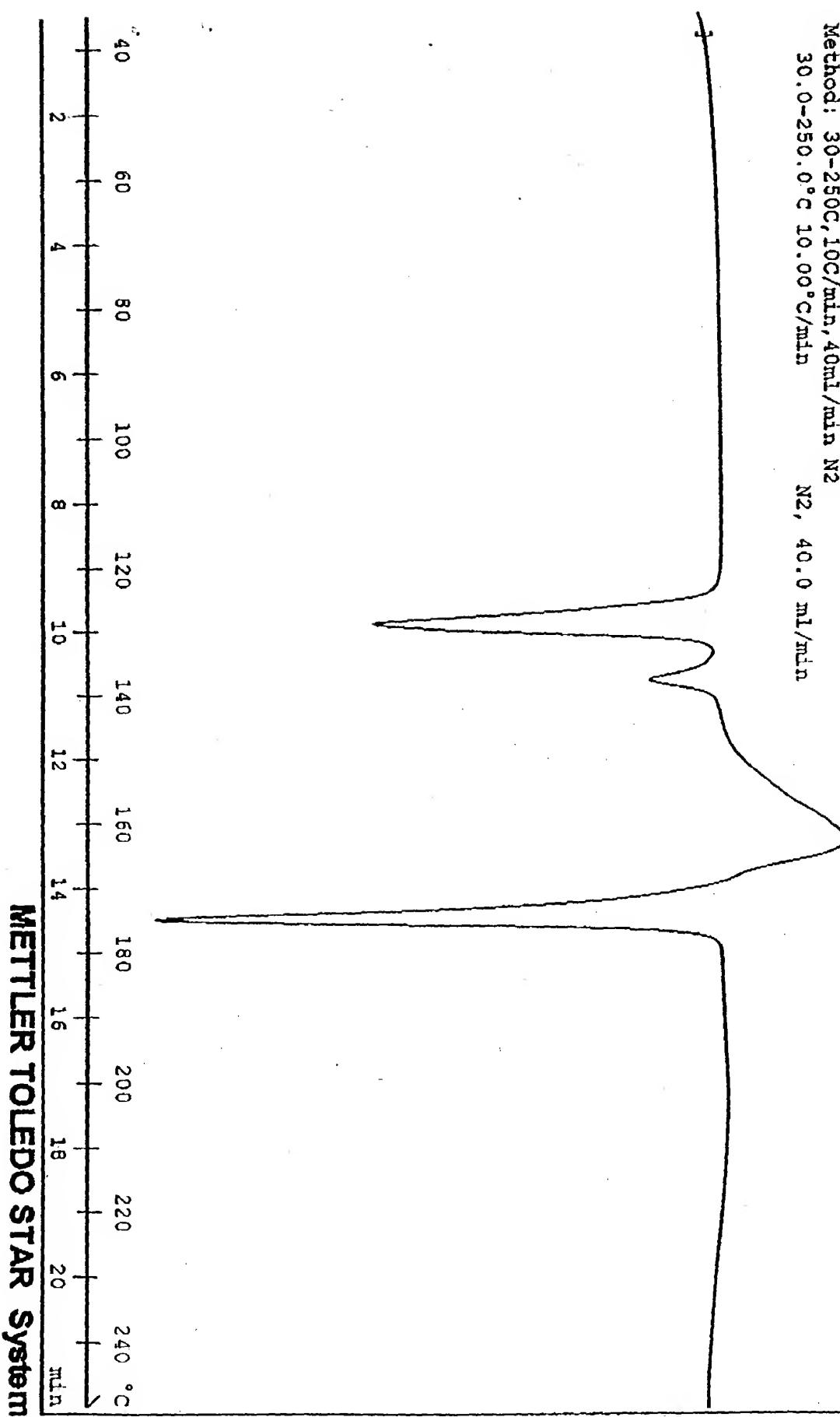
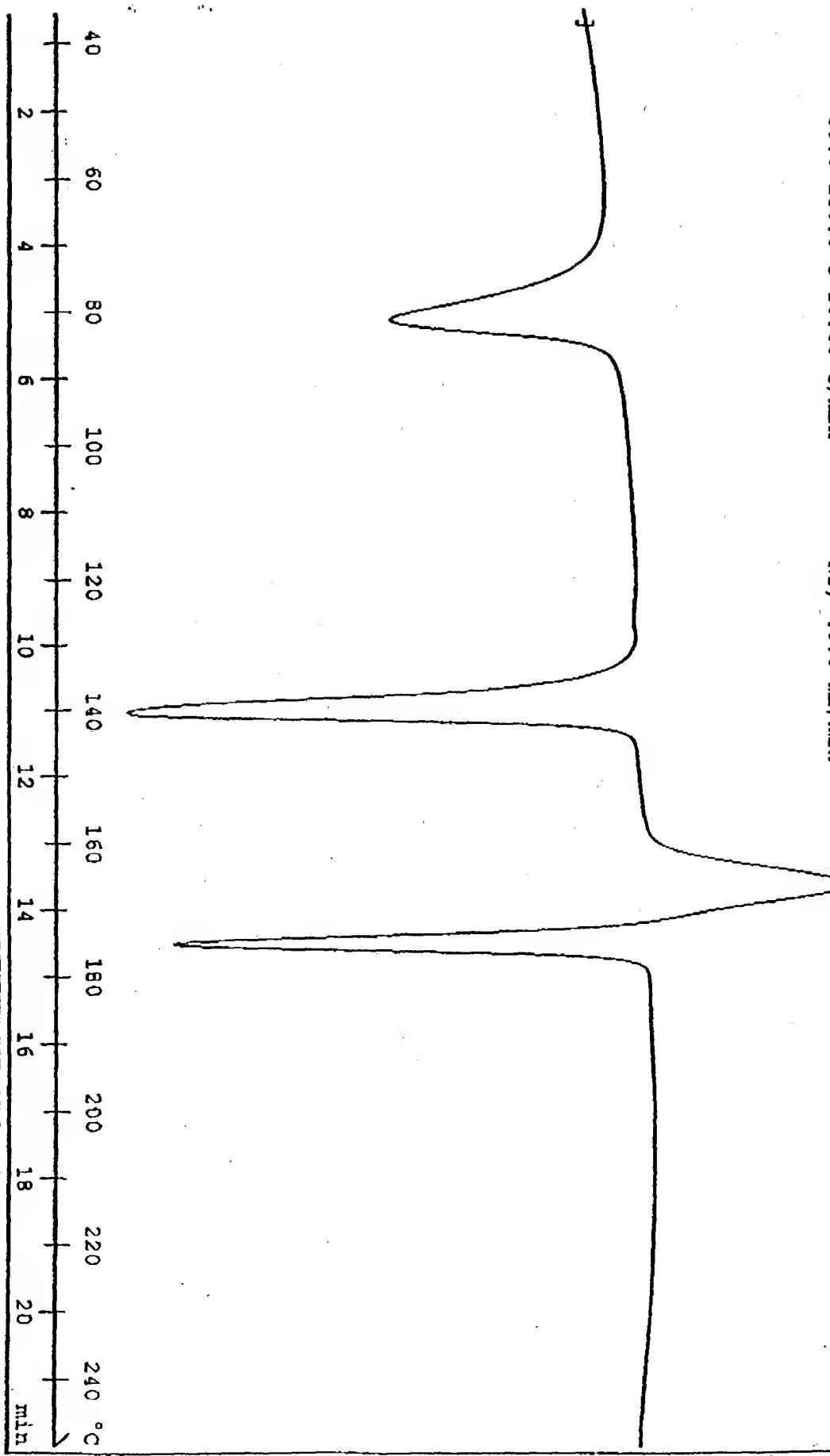


FIGURE 5e5 2
Form ✓

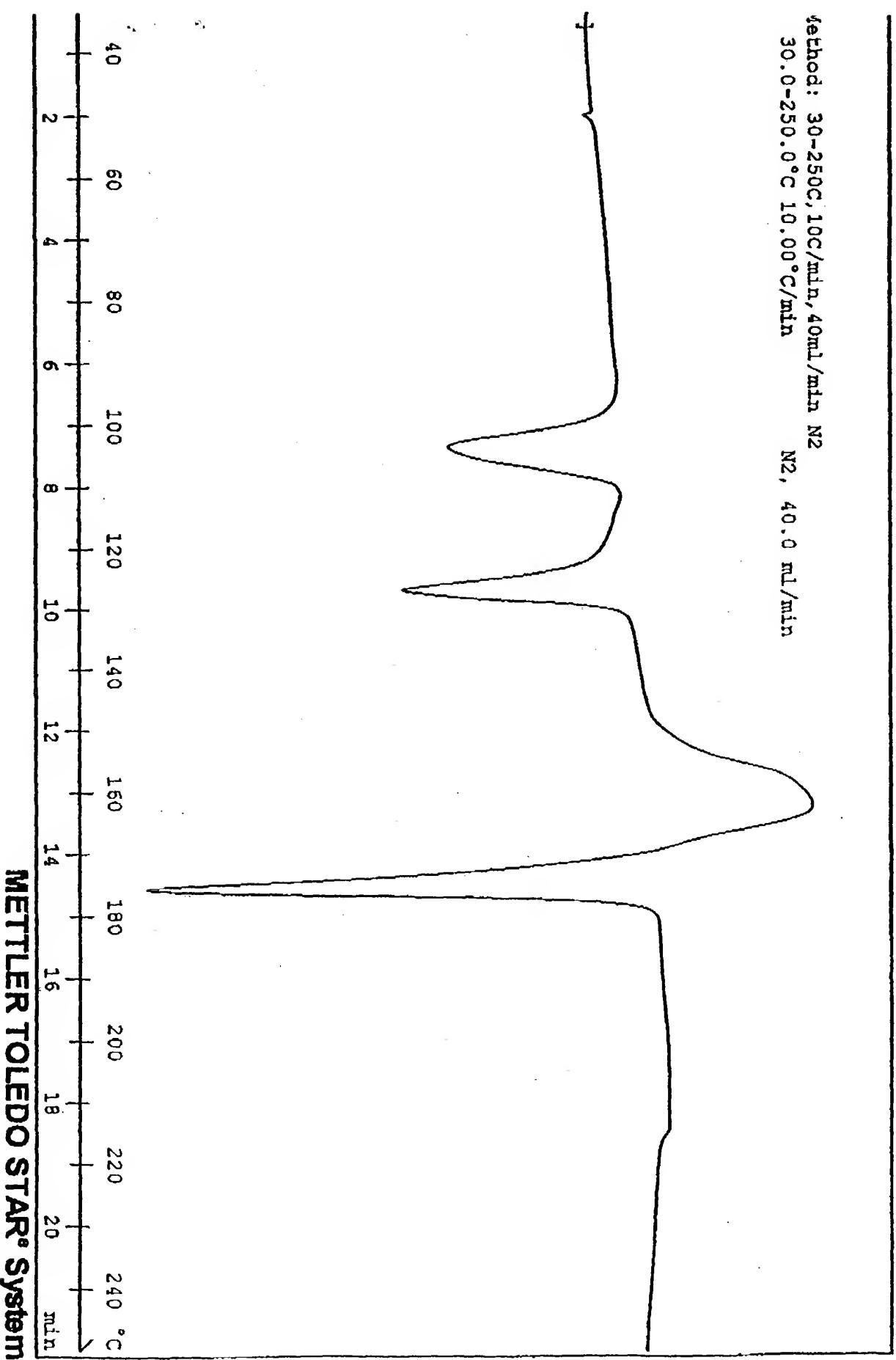
Method: 30-250C, 10C/min, 40ml/min N2
30.0-250.0 °C 10.00 °C/min
N2, 40.0 ml/min



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FIGURE 52-53
Form Y (chloroform solvent)

Method: 30-250°C, 10°C/min, 40mL/min N₂
30.0-250.0°C 10.00°C/min N₂, 40.0 mL/min



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FIGURE 54
 γ (dichloromethane solvate)

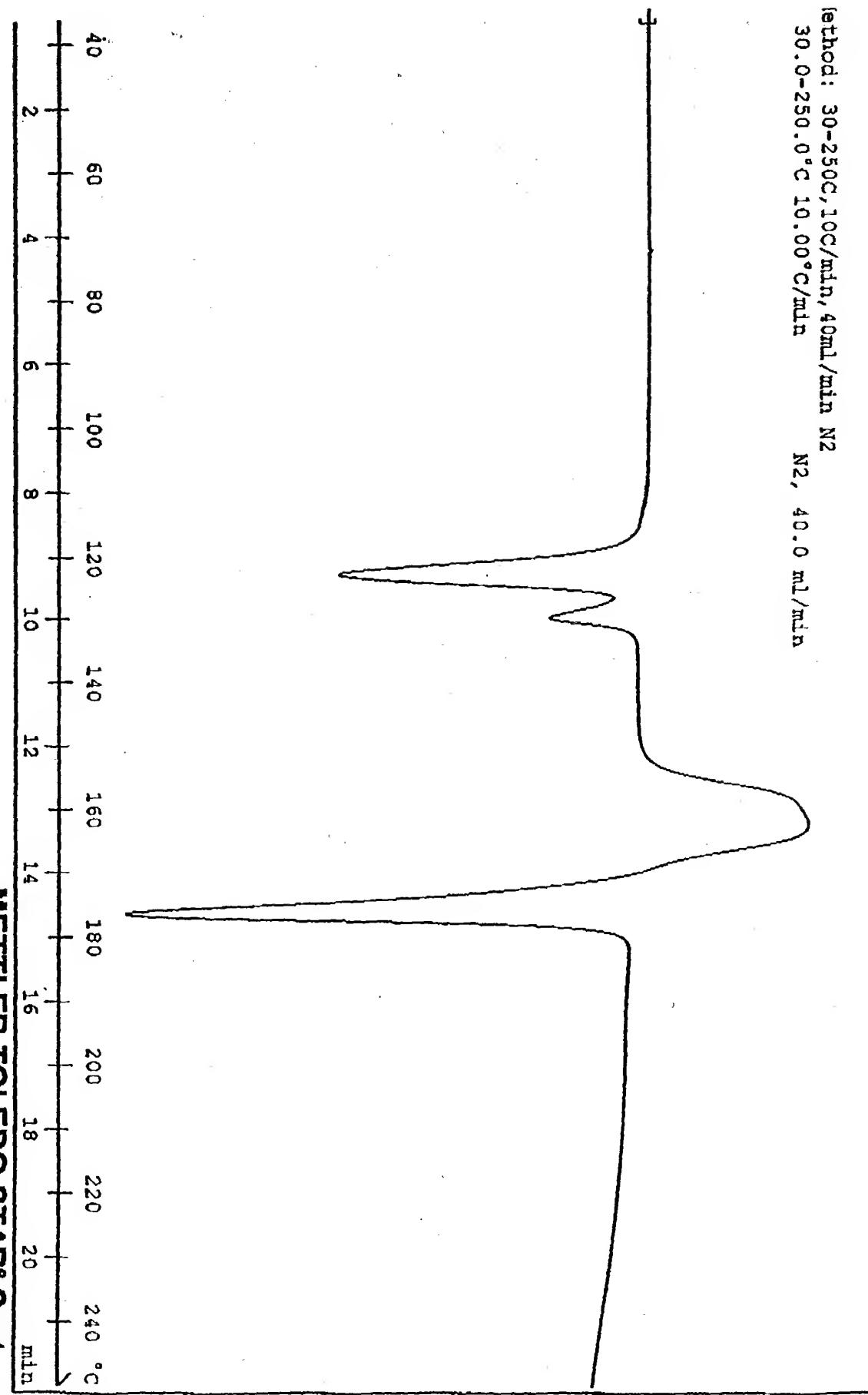


Figure 27 - Nørgaard Form Z
55

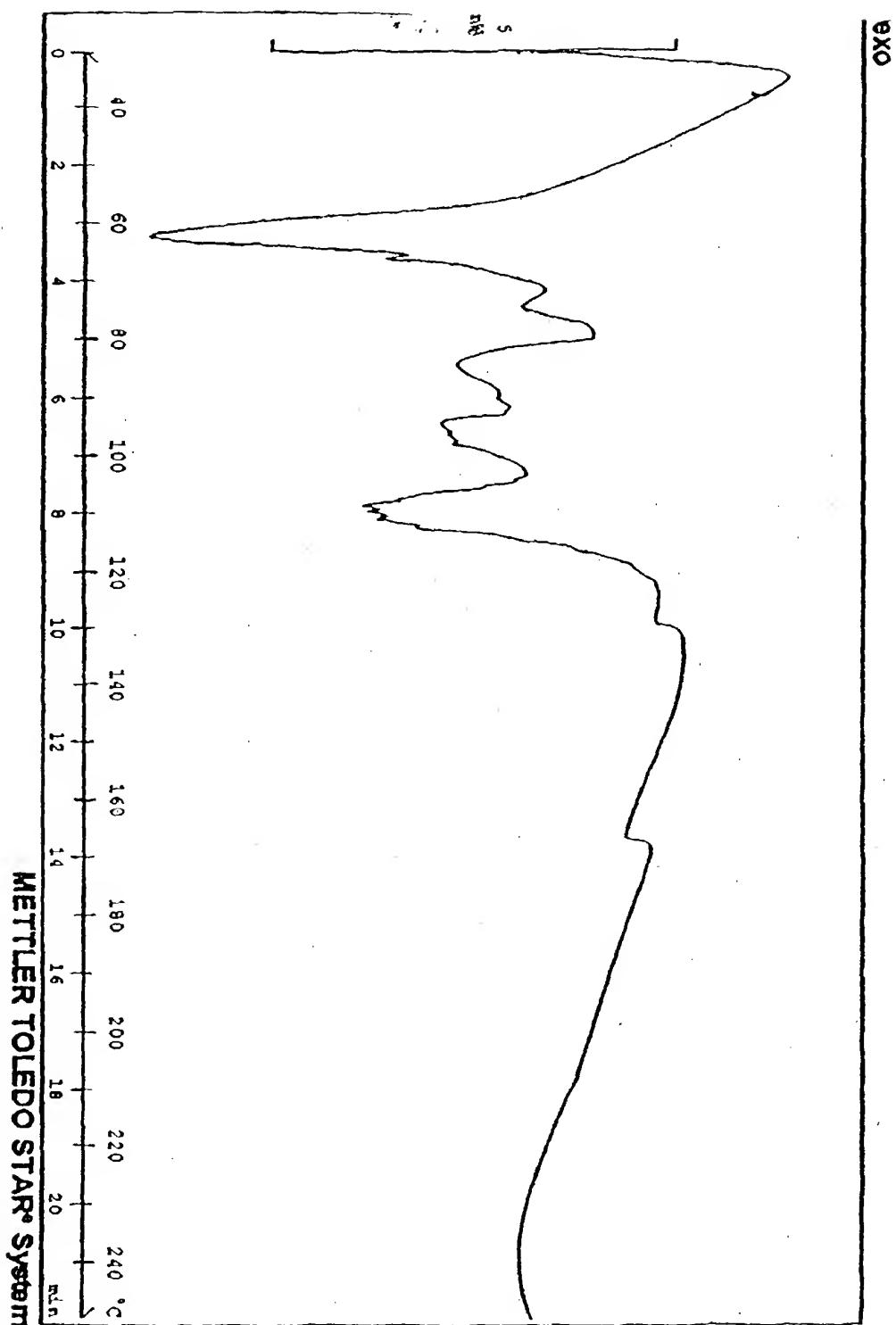
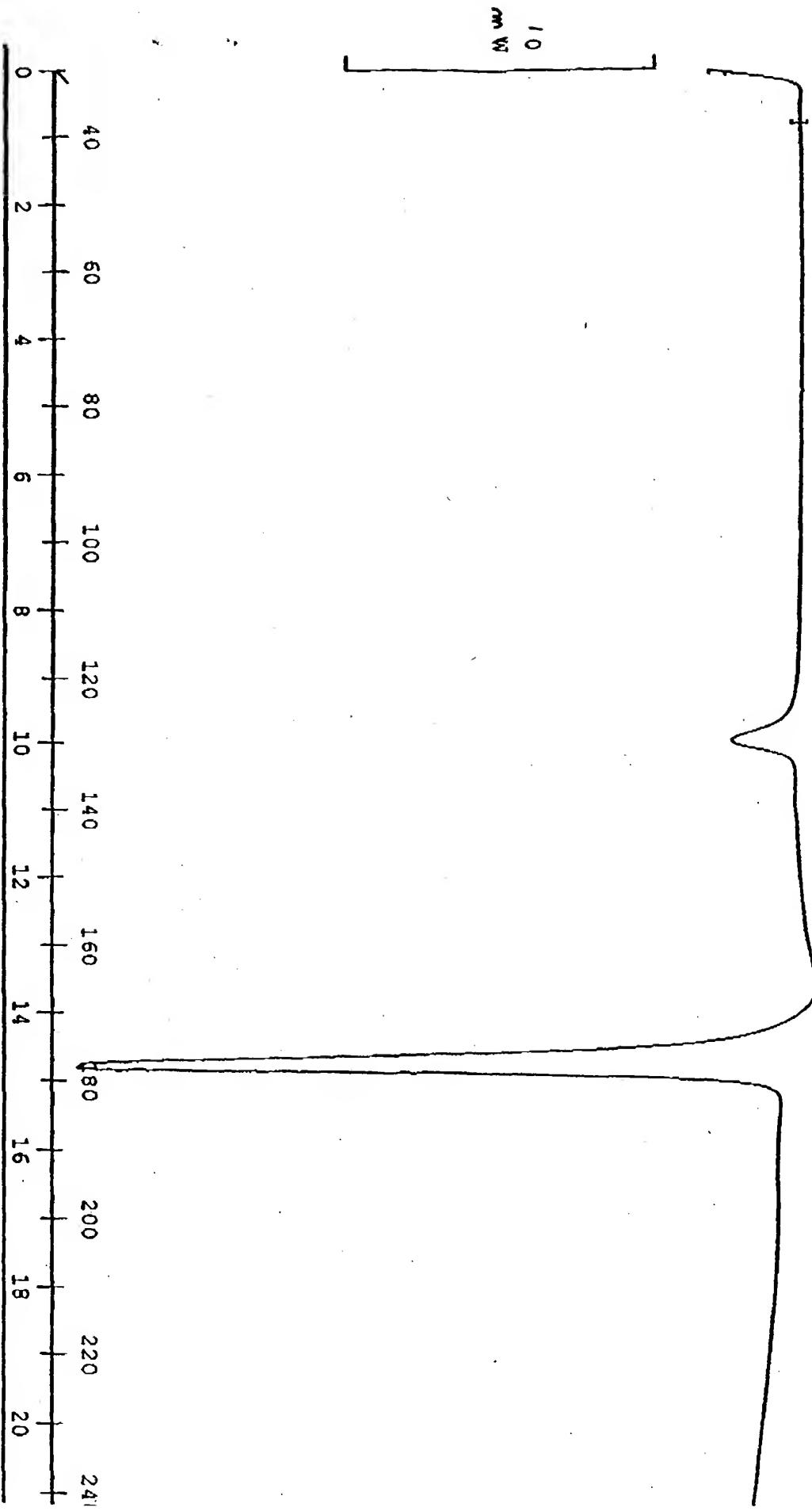


FIGURE 5-6
Form 256

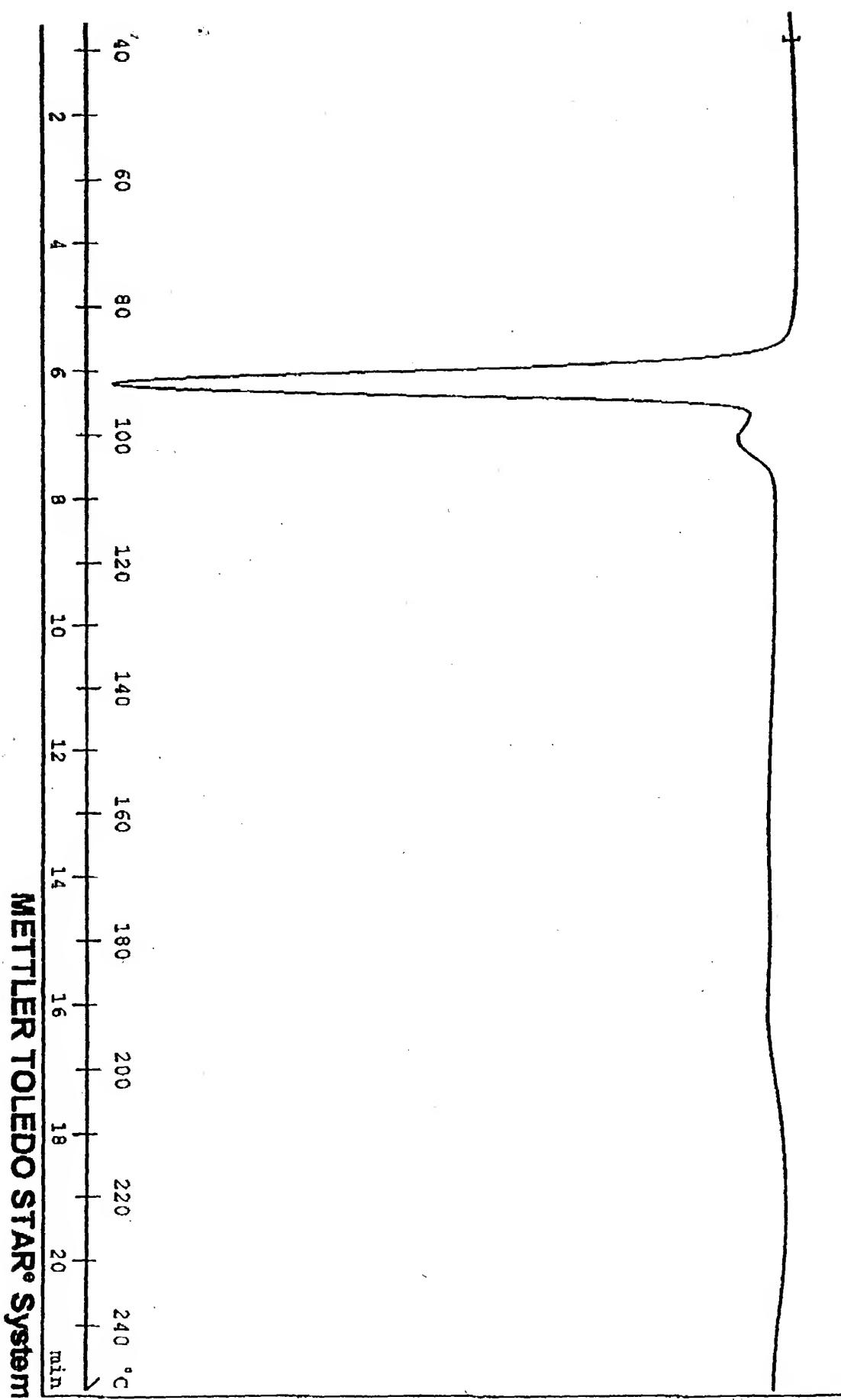
Method: 30-250°C, 10°C/min, 40mL/min N₂
 30°.0-250.0°C 10.00°C/min N₂, 40.0 mL/min



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Figure 5
Form Beta

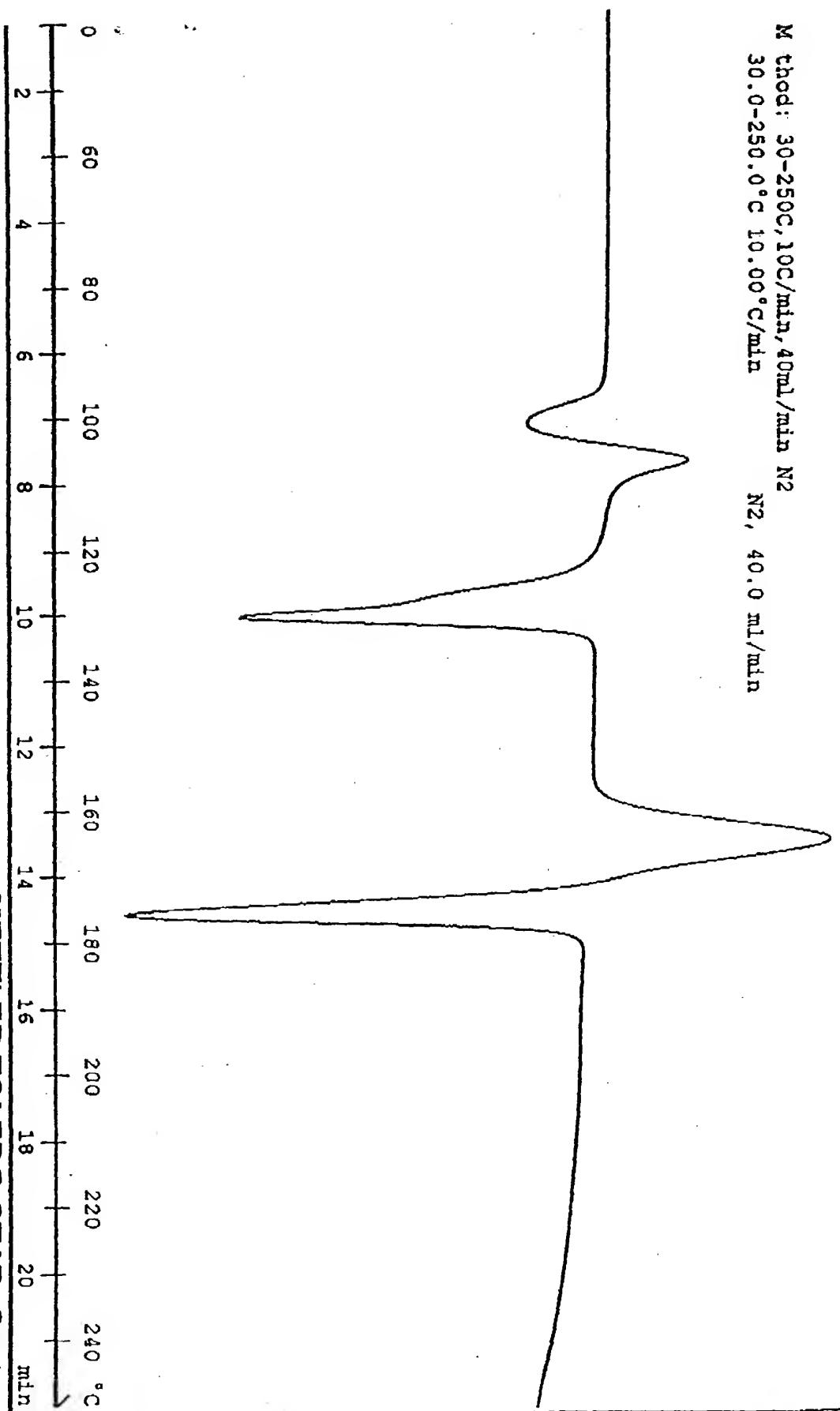
Method: 30-250C, 10C/min, 40ml/min N2
30.0-250.0°C 10.00°C/min N2, 40.0 ml/min



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FIGURE 55-58
Form Delta

Method: 30-250°C, 10°C/min, 40mL/min N₂
30.0-250.0°C 10.00 °C/min
N₂, 40.0 mL/min



- Delta

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Form-Epsilon FIGURE 56-59

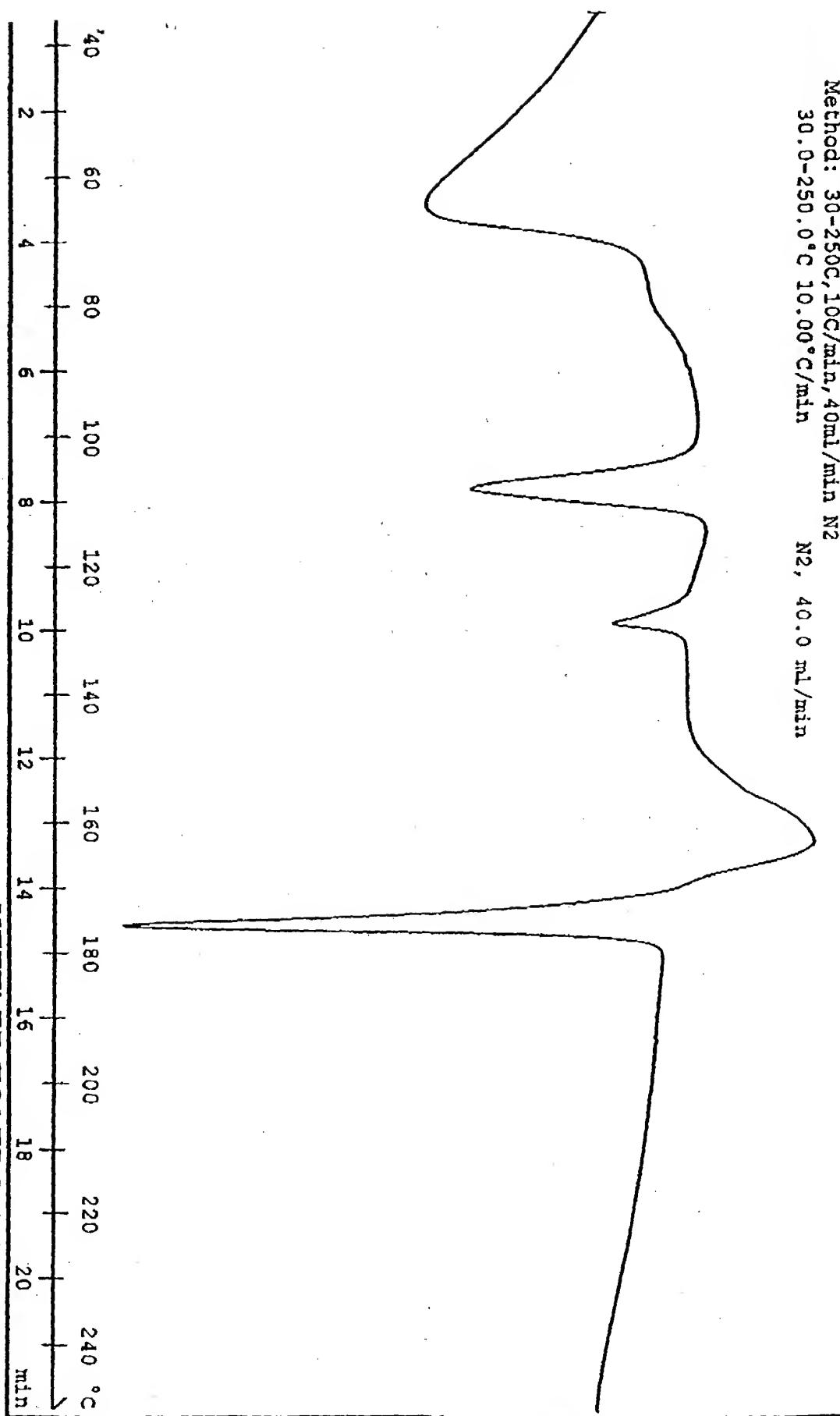
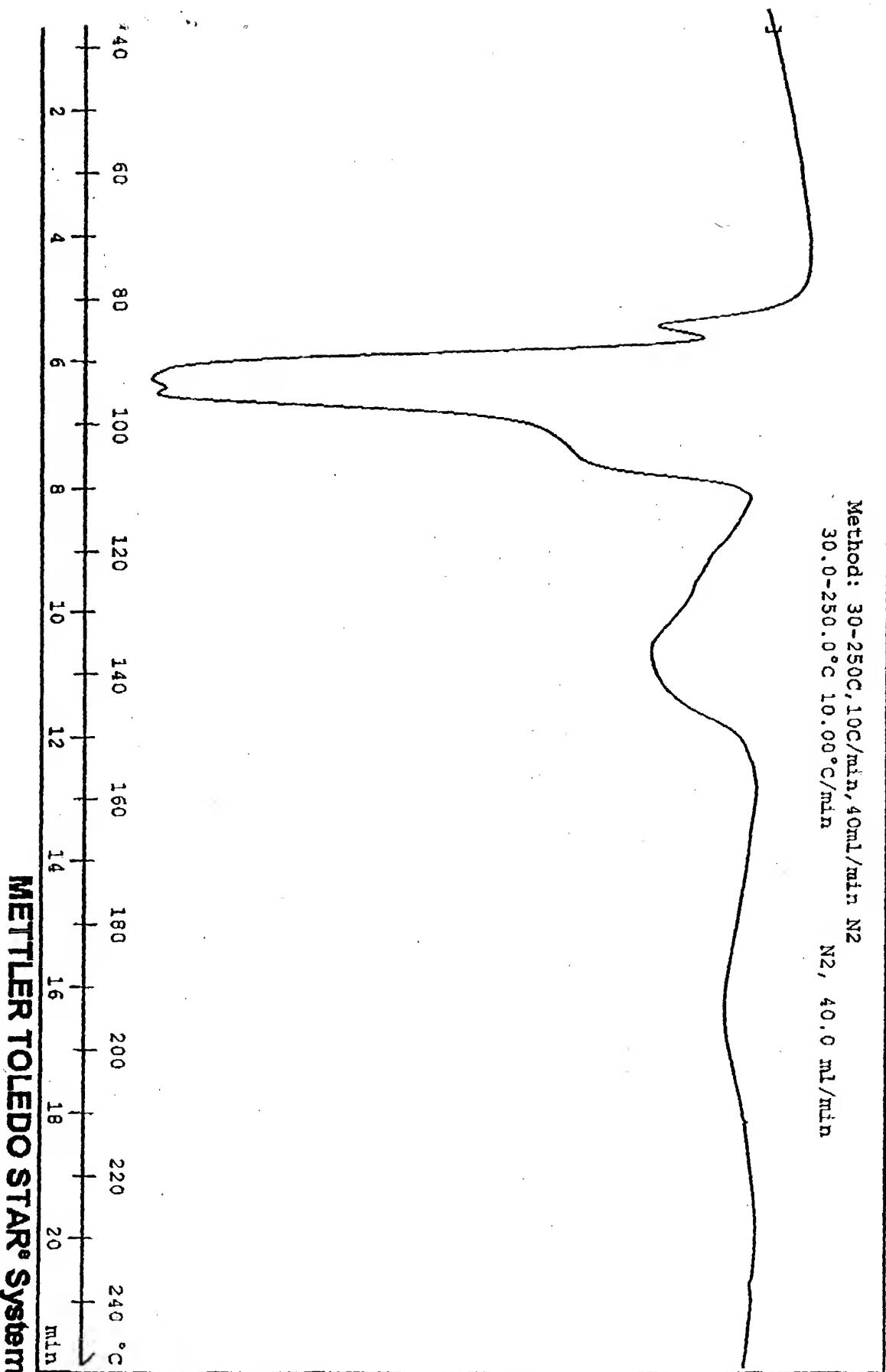


FIGURE 60

Form  Gamma

Method: 30-250°C, 10°C/min, 40mL/min N₂
30.0-250.0°C 10.00°C/min N₂, 40.0 mL/min

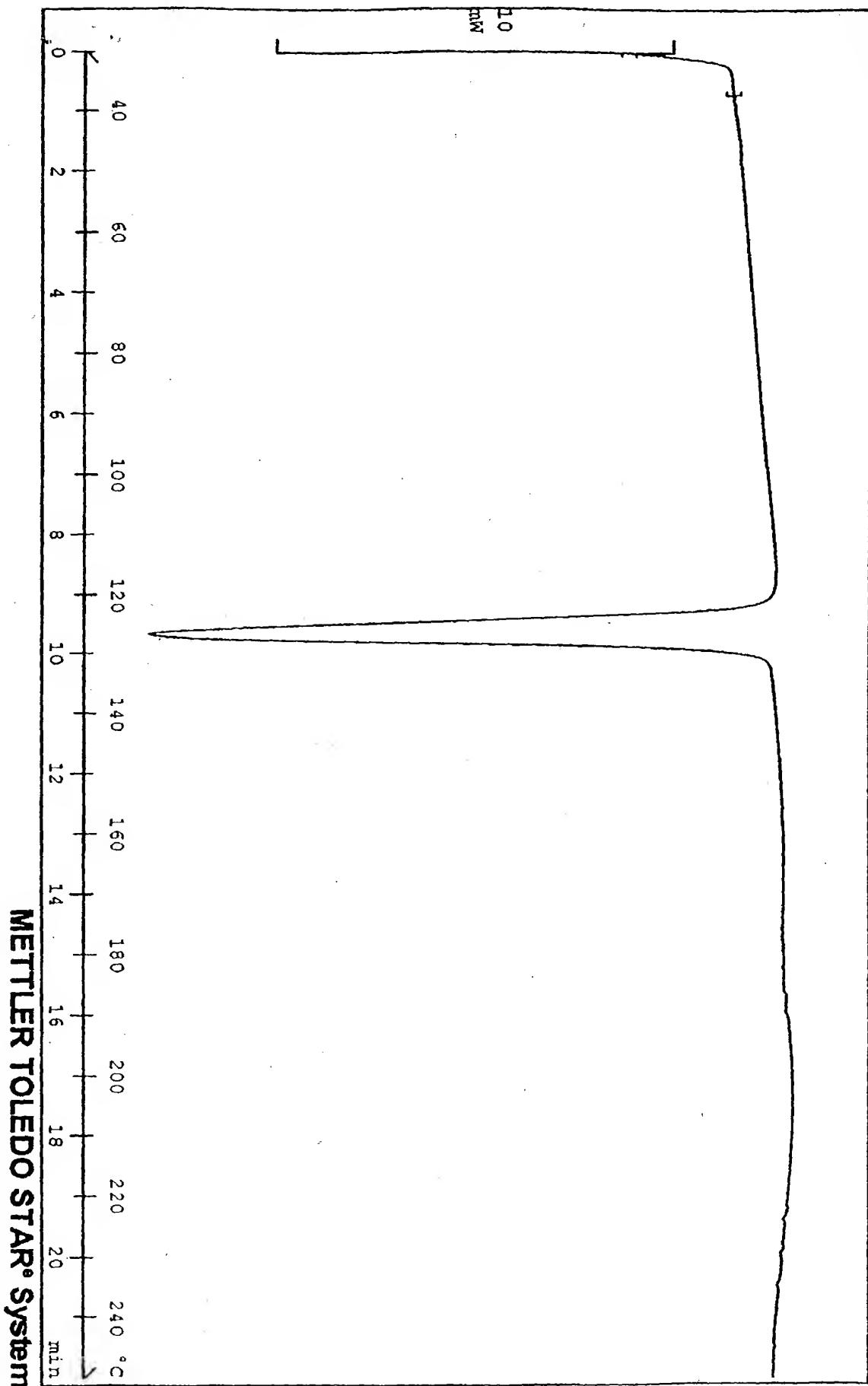


METTLER TOLEDO STAR® System

FIGURE 58 61

Form Sigma

exo



Form \sim (5)

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FIGURE 62

Form Theta

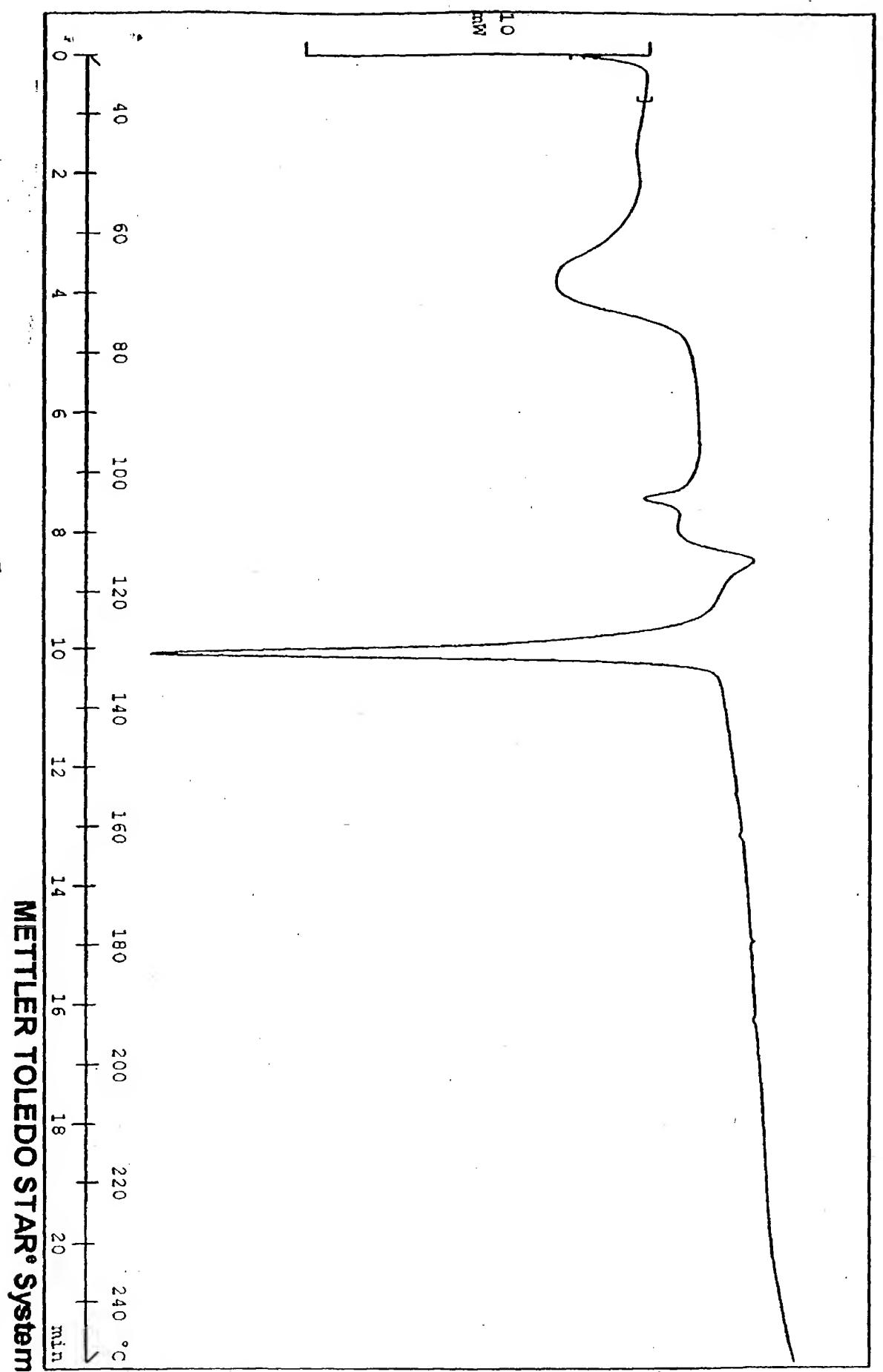


Figure 63

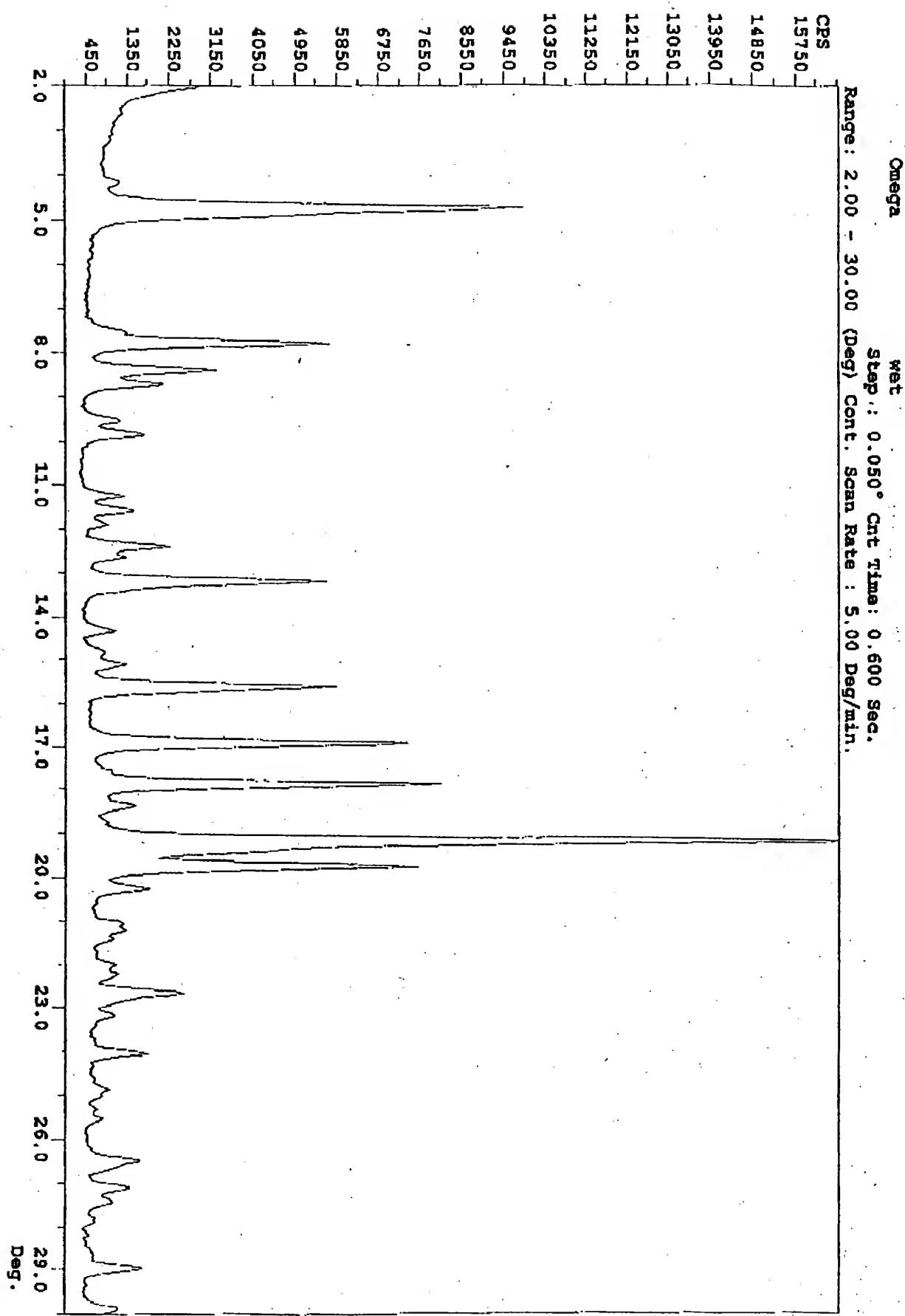


Figure 64

Comparison between the impurity profile of Nateglinide crystallized in $\text{IPA-H}_2\text{O}$ and Nateglinide crystallized in Methanol- H_2O

Sample No	Solvent	Impurity profile by RRT [% w/w]								
		D-PA (0.23)	(0.25)	(0.46)	(0.80)	Ipcha (0.89)	Dimer (1.38)	Methyl Ester (1.51)	(1.76)	Isopropyl Ester (2.3)
RL-2155/1	Methanol- H_2O	<0.01		0.02	<0.01	0.03	0.02	2.91	0.04	
RL-2163/4	$\text{IPA-H}_2\text{O}$	<0.01		0.04	0.02	0.02	0.01	0.03	0.02	

Note: D-PA means D-Phenyl Alanine

Ipcha means Iso propyl cyclohexyl carboxylic acid

Both are the starting materials of the product

(-)-N-[(trans-4-isopropyl cyclohexane)carbonyl]-D-phenylalanine